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Attorneys for Plaintiffs

**UNITED STATES DISTRICT COURT
EASTERN DISTRICT OF CALIFORNIA**

AFAB Industrial Services, Inc., a
Pennsylvania Corporation; NewBridge
Global Ventures, Inc., a Delaware
Corporation; EcoExtraction LLC, a
Louisiana Limited Liability Company; and
CleanWave Labs, LLC, a Nevada Limited
Liability Company

Plaintiffs,

v.

Apothio, LLC, a Colorado Limited
Liability Company; Trent Jones; and
Apothio Bakersfield, LLC, a Nevada
Limited Liability Company,

Defendants.

Case No.

COMPLAINT FOR:

**I. PATENT INFRINGEMENT (35
U.S.C. §271) (US 10,011,804)**

**II. PATENT INFRINGEMENT (35
U.S.C. §271) (US 9,469,548)**

**III. PATENT INFRINGEMENT
(35 U.S.C. §271) (US 8,430,968)**

**IV. PATENT INFRINGEMENT
(35 U.S.C. §271) (US 7,507,014)**

**V. PATENT INFRINGEMENT
(35 U.S.C. §271) (US 6,627,784)**

**VI. PATENT INFRINGEMENT
(35 U.S.C. §271) (US 10,220,365)**

**VII. PATENT INFRINGEMENT
By Inducement (35 U.S.C. § 271(b))**

DEMAND FOR JURY TRIAL

1 Plaintiffs AFAB Industrial Services, Inc., a Pennsylvania Corporation
2 (“AFAB”); NewBridge Global Ventures, Inc., a Delaware Corporation
3 (“NEWBRIDGE”); EcoXtraction, LLC, a Louisiana Limited Liability Company
4 (“ECO”), and CleanWave Labs, LLC, a Nevada Limited Liability Company
5 (“CleanWave”) (collectively “Plaintiffs”) hereby allege the following as their
6
7 Complaint against Defendants Apothio, LLC, a Colorado Limited Liability
8 Company (“APOTHIO”); Trent Jones (“JONES”), an individual; and Apothio
9 Bakersfield, LLC, a Nevada Limited Liability Company (“APOTHIO
10 BAKERSFIELD”) (collectively “Defendants”).
11
12

13 **JURISDICTION AND VENUE**

14
15 1. This action arises under the United States Patent Act, codified at 35
16 U.S.C. § 1 *et seq.*, and in particular, 35 U.S.C. §§ 271 and 281-285.

17 2. This Court has original jurisdiction over the subject matter of this
18
19 action under 28 U.S.C. §§ 1331 and 1338(a).

20 3. This Court has personal jurisdiction over Defendants because, upon
21
22 information and belief, Defendants have engaged and continue to engage in
23
24 significant business activity within the State of California and within this judicial
District.

25 4. Upon information and belief, Defendants have also caused and
26
27 continue to cause injury to Plaintiffs’ within the State of California and within this
28

1 judicial district by virtue of their infringement of the Patents, as hereinafter
2 defined.

3
4 5. As a result of their actions, Defendants have sufficient personal
5 contacts with the State of California and this judicial District, which subjects them
6 to the general and specific jurisdiction of Plaintiffs' claims.

7
8 6. Venue is proper under 28 U.S.C. §§ 1391(b), 1391(c) and 1400(b) in
9 this District because, a substantial part of the events or omissions giving rise to
10 Plaintiffs' claims occurred in this District, and the injuries caused to Plaintiffs and
11 the Patents occurred in this District.

12
13 **NATURE OF THE ACTION**

14
15 7. This is an action for patent infringement against Defendants for their
16 infringement of the following United States Patents: No. 10,011,804
17 ("the '804 Patent"), No. 9,469,548 ("the '548 Patent"), No. 8,430,968
18 ("the '968 Patent"), No. 7,507,014 ("the '014 Patent"), No. 6,627,784
19 ("the '784 Patent"), and No. 10,220,365 ("the '365 Patent") (collectively,
20 the "Patents").
21

22
23 **THE PARTIES**

24
25 8. Plaintiff AFAB Industrial Services, Inc. is a Pennsylvania Corporation
26 in good standing with its principal place of business located in Bensalem,
27 Pennsylvania.
28

1 9. Plaintiff, NewBridge Global Ventures, Inc., is a Delaware Corporation
2 in good standing with its principal place of business in Alameda, California.
3

4 10. Plaintiff EcoXtraction, LLC is a Louisiana Limited Liability Company
5 in good standing with its principal place of business in Lafayette, Louisiana.
6

7 11. Plaintiff CleanWave Labs, LLC is a Nevada Limited Liability
8 Company in good standing with its principal place of business in Alameda,
9 California.
10

11
12 12. Defendant Apothio, LLC is a Colorado Limited Liability Company
13 with its principal place of business in Peru, Indiana. It is registered with the
14 California Secretary of State to conduct business in the State of California.
15

16 13. Defendant Trent Jones, an individual, is a resident of the State of
17 Indiana and is the, or one of the, Managing Members of Defendants Apothio, LLC
18 and Apothio Bakersfield, LLC.
19

20
21 14. Defendant Apothio Bakersfield, LLC is a Nevada Limited Liability
22 Company with its principal place of business in the County of Kern, State of
23 California.
24
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FACTUAL BACKGROUND

15. Plaintiffs allege, as if fully set forth herein, the allegations contained in paragraphs 1- 14 above.

16. On or about May 2, 2019, NEWBRIDGE and APOTHIO entered in an agreement to form APOTHIO BAKERSFIELD, a business arrangement whereby APOTHIO would provide industrial hemp for processing by NEWBRIDGE in order to produce cannabidiol, commonly referred to as CBD oil.

17. The concept was to take local hemp provided by APOTHIO and apply a new, patented, and proprietary technology exclusively owned and/or licensed to and being developed by NEWBRIDGE that would process the hemp in a continuous oil extraction process using water as a medium, rather than batch extraction using ethanol and CO2 as is currently used today.

18. In connection with the creation of this new entity, NEWBRIDGE and APOTHIO also entered into a Non-Circumvention, Non-Disclosure and Confidentiality Agreement.

19. In order to facilitate processing, NEWBRIDGE brought in a partner, AFAB.

20. NEWBRIDGE also brought in technology from another of its business partners, ECO.

1 21. NEWBRIDGE and ECO created a joint venture known as
2 CLEANWAVE. NEWBRIDGE, ECO, AFAB and CLEANWAVE either loaned,
3 purchased or leased all of the processing equipment and technology to be used to
4 process the hemp that was to be provided by APOTHIO (hereinafter, the
5 “Equipment”).
6

7
8 22. NEWBRIDGE owns or has been licensed to use, and will continue to
9 own or be licensed to use, all of the Equipment and licenses for the technology to
10 have been used by APOTHIO BAKERSFIELD, including, but not limited to, all
11 Patents, all extraction equipment and technologies, and all testing equipment and
12 technology.
13
14

15 23. The Patents owned or exclusively licensed to NEWBRIDGE were to
16 be licensed to APOTHIO BAKERSFIELD, but an agreement has not, as of the
17 date of the filing of this Complaint, been reached.
18
19

20 24. None of the Equipment is owned by any Defendant in this matter. The
21 bulk of the Equipment provided to NEWBRIDGE was on lease or loan from third
22 party owners of the Equipment. Those owners have now informed NEWBRIDGE
23 that NEWBRIDGE needs to return that Equipment. Both CLEANWAVE and
24 others have demanded return of their Equipment located at the APOTHIO Los
25 Osos Facility, in Kern County, CA.
26
27
28

1 25. In order to set up the processing facility, a property, located at 580 S.
2 Derby St. within the City of Arvin, County of Kern State of California was
3 acquired.
4

5 26. This facility is the “Los Osos Facility.” Plaintiffs are informed and
6 believe, and thereupon allege, that the Los Osos Facility was, and at all times
7 relevant to this matter was, owned by Lehr Brothers Inc. (“LEHR”).
8
9

10 27. The Operating Agreement for APOTHIO BAKERSFIELD states that
11 APOTHIO is in the process of acquiring real estate necessary for the extraction
12 and processing of the hemp crop being grown by APOTHIO Plaintiffs are
13 informed and believe, and thereupon allege, that the real estate in question was the
14 Los Osos Facility. Once the Los Osos Facility was acquired, the Operating
15 Agreement provides that APOTHIO BAKERSFIELD will, “enjoy a long-term
16 lease to a portion of these facilities as mutually agreed between APOTHIO and
17 APOTHIO BAKERSFIELD.”
18
19
20

21 28. Plaintiffs have been informed that LEHR and APOTHIO did enter into
22 a lease for the Los Osos Facility.
23

24 29. By letters dated August 30, 2019, Louisiana Counsel for Plaintiff ECO
25 demanded that Defendants JONES and APOTHIO release the Equipment of
26 Plaintiffs, but said Defendants refused.
27
28

1 30. On September 9, 2019, Plaintiffs ECO and CLEANWAVE filed suit
2 against all Defendants and LEHR in California State Court on several state causes
3 of action demanding, among other things, that Defendants release the Equipment,
4 but Defendants again, refused.
5

6
7 **THE PATENTS**
8

9 31. ECO is the record owner of United States Patent No. 10,011,804.
10 Attached hereto as Exhibit “A” is a true and correct copy of the ’804 Patent.
11

12 32. Hydro Dynamics, Inc. of Rome, GA is the record owner of the
13 following United States Patents: (i) No. 9,469,548; (ii) No. 8,430,968;
14 (iii) No. 7,507,014; (iv) No. 6,627,784; and (v) No. 10,220,365. Attached hereto
15 as Exhibits “B” – “F” are true and correct copies of the aforementioned patents.
16

17
18 33. By License Agreement dated March 11, 2018, Hydro Dynamics, Inc.
19 granted ECO a license to the ’968 Patent and the ’784 Patent, including the right to
20 sue infringers of same.
21

22 34. By License Agreement dated April 12, 2019, Hydro Dynamics, Inc.
23 granted NEWBRIDGE a license to the ’365 Patent, the ’548 Patent, the ’014
24 Patent, and the ’784 Patent, including the right to sue infringers of same.
25

26
27 35. By Assignment and License Agreement dated February 14, 2019 and
28 Asset Purchase Agreement dated February 14, 2019, ECO assigned and transferred

1 to NEWBRIDGE and CLEANWAVE its rights under the License Agreement with
2 Hydro Dynamics, Inc., including joint rights to sue infringers.
3

4 **DEMAND FOR CESSATION OF INFRINGEMENT AND RETURN OF**
5 **EQUIPMENT ALLOWING INFRINGEMENT**

6 36. On August 11, 2019, JONES told others he had changed the locks at
7 the Los Osos Facility and was preparing to lock NEWBRIDGE and AFAB out of
8 the facility.
9

10 37. By letter of September 20, 2019, Counsel for Plaintiffs herein
11 demanded that Defendants return the Equipment and intellectual property to
12 Plaintiffs.
13

14 38. By letter of September 23, 2019, Counsel for Defendant APOTHIO
15 acknowledged that said Equipment “have been set aside pending resolution of the
16 claims between the parties and have not been used,” but has failed to identify any
17 legal basis whatsoever for this refusal to return the Equipment which can easily be
18 used to infringe the Patents.
19
20
21

22 **DEFENDANTS’ INFRINGEMENT**
23

24 39. Defendant APOTHIO is in the business of growing, processing and
25 selling hemp. As such, it requires means to process the hemp and extract
26 marketable CBD oils.
27
28

1 40. The extraction process embodied in and protected by the Patents is
2 efficient, economical, and extremely useful to a business such as Defendant,
3 APOTHIO' business.
4

5 41. Defendants JONES and APOTHIO have already deceived
6 NEWBRIDGE and violated the APOTHIO BAKERSFIELD Operating Agreement
7 (5/2/2019) and Non-Circumvention, Non-Disclosure and Confidentiality
8 Agreement (5/7/2019) and illegally locked Plaintiffs out and seized Plaintiffs'
9 Equipment embodying the Patents.
10
11

12 42. Even if Defendants have not yet "used" the Equipment covered by the
13 Patents, the circumstantial evidence is that they have threatened and/or
14 contemplated use and that is actionable.
15
16

17 43. Defendants are also currently advertising on the Apothio.net website
18 that "Apothio is developing cutting edge technology that replaces toxic and
19 volatile solvents from the extraction process, and replaces it with water" and that
20 "Apothio uses proprietary whole plant extraction technology that uses water as the
21 solvent, which produces a safe and compliant water soluble extract." A true and
22 correct copy of a print-out of the Apothio.net website is attached hereto as
23 Exhibit "G."
24
25
26

27 44. Upon information and belief, APOTHIO has paid since on or before
28 July 22, 2019 and continues to pay rent of \$10,000, plus expenses, per month for

1 the Los Osos Facility. As there is no other function for the Los Osos Facility other
2 than for use of Plaintiffs' Equipment for hemp extraction, this is further evidence
3 that Defendants are infringing and/or about to infringe the Patents.
4

5
6 45. Under these circumstances, unsworn, bald claims by Defendant
7 APOTHIO's Counsel, that APOTHIO has not used the Equipment - has not
8 infringed the Patents - and will not do so is insufficient and unreliable as a matter
9 of fact and law.
10

11
12 46. Upon information and belief, and logic and experience with
13 Defendants, Defendants have, are now and/or will likely use Plaintiff's Equipment
14 and technology without Plaintiffs' authorization and thus have already infringed,
15 are presently infringing and/or are very likely to infringe the Patents.
16

17
18 47. Even if Defendants have not actually "used" the Equipment to extract
19 CBD oil from hemp, their claim to rightfully continue to possess same is a claim of
20 right to put it into service which is a "right to use," a term interpreted broadly by
21 Courts, and thus constitutes infringement.
22

23
24 48. Defendants' legally baseless refusal to return, and continued
25 possession, constitutes threatened and/or contemplated use as it is held for
26 purposes of profit and thus *is* infringement.
27
28

1 49. Upon information and belief, Defendants have repaired and/or
2 maintained the Equipment and such constitutes use and infringement.

3
4 50. Defendants, as unauthorized users, are direct, willful infringers, and/or
5 contributing infringers, as well as joint tortfeasors.

6
7 **COUNT I - INFRINGEMENT OF THE '804 PATENT**
8 **(Against all Defendants)**

9 51. Plaintiff realleges and incorporates by reference paragraphs 1 through
10 50 of this Complaint as though fully set forth herein.

11 52. The claims of the '804 Patent are presumed valid pursuant to
12 35 U.S.C. § 282.

13
14 53. The Equipment currently within Defendants' possession, custody and
15 control, contains the technology and/or inventions outlined in the '804 Patent.

16
17 54. Upon information and belief, Defendants have been and are currently
18 infringing one or more claims of the '804 Patent, either literally or under the
19 doctrine of equivalents, by using, without license or authority, the technology
20 and/or inventions covered by one or more of the claims of the '804 Patent,
21 including at least one claim of the '804 Patent.

22
23 55. As a result of Defendants' infringement of the '804 Patent, Plaintiff
24 has been damaged to an extent not yet determined.

1 56. Plaintiff is entitled to injunctive relief and monetary damages adequate
2 to compensate it for infringement by Defendants of the '804 Patent, together with
3 interest and costs.
4

5 **COUNT II - INFRINGEMENT OF THE '548 PATENT**
6 **(Against all Defendants)**

7 57. Plaintiff realleges and incorporates by reference paragraphs 1 through
8 56 of this Complaint as though fully set forth herein.
9

10 58. The claims of the '548 Patent are presumed valid pursuant to
11 35 U.S.C. § 282.
12

13 59. The Equipment currently within Defendants' possession, custody and
14 control, contains the technology and/or inventions outlined in the '548 Patent.

15 60. Upon information and belief, Defendants have been and are currently
16 infringing one or more claims of the '548 Patent, either literally or under the
17 doctrine of equivalents, by using, without license or authority, the technology
18 and/or inventions covered by one or more of the claims of the '548 Patent,
19 including at least one claim of the '548 Patent.
20
21

22 61. As a result of Defendants' infringement of the '548 Patent, Plaintiff
23 has been damaged to an extent not yet determined.
24

25 62. Plaintiff is entitled to injunctive relief and monetary damages adequate
26 to compensate it for infringement by Defendants of the '548 Patent, together with
27 interest, costs and all attorneys' fees.
28

COUNT III - INFRINGEMENT OF THE '968 PATENT
(Against all Defendants)

63. Plaintiff realleges and incorporates by reference paragraphs 1 through 62 of this Complaint as though fully set forth herein.

64. The claims of the '968 Patent are presumed valid pursuant to 35 U.S.C. § 282.

65. The Equipment currently within Defendants' possession, custody and control, contains the technology and/or inventions outlined in the '968 Patent.

66. Upon information and belief, Defendants have been and are currently infringing one or more claims of the '968 Patent, either literally or under the doctrine of equivalents, by using, without license or authority, the technology and/or inventions covered by one or more of the claims of the '968 Patent, including at least one claim of the '968 Patent.

67. As a result of Defendants' infringement of the '968 Patent, Plaintiff has been damaged to an extent not yet determined.

68. Plaintiff is entitled to injunctive relief and monetary damages adequate to compensate it for infringement by Defendants of the '968 Patent, together with interest, costs and all attorneys' fees.

COUNT IV - INFRINGEMENT OF THE '014 PATENT
(Against all Defendants)

69. Plaintiff realleges and incorporates by reference paragraphs 1 through 68 of this Complaint as though fully set forth herein.

1 70. The claims of the '014 Patent are presumed valid pursuant to
2 35 U.S.C. § 282.

3
4 71. The Equipment currently within Defendants' possession, custody and
5 control, contains the technology and/or inventions outlined in the '014 Patent.

6 72. Upon information and belief, Defendants have been and are currently
7 infringing one or more claims of the '014 Patent, either literally or under the
8 doctrine of equivalents, by using, without license or authority, the technology
9 and/or inventions covered by one or more of the claims of the '014 Patent,
10 including at least one claim of the '014 Patent.

11
12 73. As a result of Defendants' infringement of the '014 Patent, Plaintiff
13 has been damaged to an extent not yet determined.

14
15 74. Plaintiff is entitled to injunctive relief and monetary damages adequate
16 to compensate it for infringement by Defendants of the '014 Patent, together with
17 interest, costs and all attorneys' fees.

18
19
20 **COUNT V - INFRINGEMENT OF THE '784 PATENT**
21 **(Against all Defendants)**

22 75. Plaintiff realleges and incorporates by reference paragraphs 1 through
23 74 of this Complaint as though fully set forth herein.

24 76. The claims of the '784 Patent are presumed valid pursuant to
25 35 U.S.C. § 282.

26
27 77. The Equipment currently within Defendants' possession, custody and
28 control, contains the technology and/or inventions outlined in the '784 Patent.

1 78. Upon information and belief, Defendants have been and are currently
2 infringing one or more claims of the '784 Patent, either literally or under the
3 doctrine of equivalents, by using, without license or authority, the technology
4 and/or inventions covered by one or more of the claims of the '784 Patent,
5 including at least one claim of the '784 Patent.
6

7
8 79. As a result of Defendants' infringement of the '784 Patent, Plaintiff
9 has been damaged to an extent not yet determined.

10 80. Plaintiff is entitled to injunctive relief and monetary damages adequate
11 to compensate it for infringement by Defendants of the '784 Patent, together with
12 interest, costs and all attorneys' fees.
13

14 **COUNT VI - INFRINGEMENT OF THE '365 PATENT**
15 **(Against all Defendants)**

16 81. Plaintiff realleges and incorporates by reference paragraphs 1 through
17 80 of this Complaint as though fully set forth herein.
18

19 82. The claims of the '365 Patent are presumed valid pursuant to
20 35 U.S.C. § 282.
21

22 83. The Equipment currently within Defendants' possession, custody and
23 control, contains the technology and/or inventions outlined in the '365 Patent.

24 84. Upon information and belief, Defendants have been and are currently
25 infringing one or more claims of the '365 Patent, either literally or under the
26 doctrine of equivalents, by using, without license or authority, the technology
27
28

1 and/or inventions covered by one or more of the claims of the '365 Patent,
2 including at least one claim of the '365 Patent.

3
4 85. As a result of Defendants' infringement of the '365 Patent, Plaintiff
5 has been damaged to an extent not yet determined.

6
7 86. Plaintiff is entitled to injunctive relief and monetary damages adequate
8 to compensate it for infringement by Defendants of the '365 Patent, together with
9 interest, costs and all attorneys' fees.

10 **COUNT VII - PATENT INFRINGEMENT BY INDUCEMENT**
11 **(Against all Defendants)**

12
13 87. Plaintiff realleges and incorporates by reference paragraphs 1 through
14 86 of this Complaint as though fully set forth herein.

15
16 88. Defendants are aware of Plaintiffs' rights in the Patents and that the
17 technology and inventions outlined in the Patents are contained within the
18 Equipment currently within Defendants' possession, custody, and/or control.

19
20 89. Upon information and belief, notwithstanding Defendants' knowledge
21 of Plaintiffs' rights in and to the Patents, each Defendant is collectively and
22 individually, inducing the infringement of Plaintiffs' Patents by the others via the
23 acts and omissions included in the allegations above.

24
25 90. As a result of Defendants' inducement, Plaintiffs' rights in the Patents
26 are being infringed and Plaintiffs have been damaged to an extent not yet
27 determined.
28

91. Plaintiff is entitled to injunctive relief and monetary damages adequate to compensate it for infringement by Defendants, together with interest, costs and all attorneys' fees.

JURY DEMAND

Plaintiff hereby demands a jury trial on all issues appropriately triable by jury.

PRAYER FOR RELIEF

WHEREFORE, Plaintiffs, prays for judgment in their favor and against Defendants. for the following:

a. A judgment that Defendants have willfully infringed Plaintiffs' Patents;

b. An award to Plaintiffs for damages that are adequate to fully compensate it for Defendants' infringement of the Patents, together with enhanced damages, prejudgment interests and costs;

c. Preliminary injunctive relief and enjoinder of Defendants to return the equipment;

d. A permanent injunction enjoining Defendants and those in active concert or participation therewith from infringing the Patents;

e. A finding that this case is exceptional and award Plaintiffs their costs, reasonable attorneys' fees, and expenses incurred in this action;

1 f. An accounting for damages from the infringement of the
2 Patents by Defendants (and those in privity with Defendants); and

3
4 g. An award of such other and further relief, at law or in
5 equity, as the Court may deem just and proper.

6 Dated: September 25, 2019

7
8 PEPPER HAMILTON LLP

9 By: s/ Jeffrey M. Goldman

10 Jeffrey M. Goldman
11 M. Kelly Tillery (*Pro Hac Vice*
12 *application to be filed*)

13
14 *Attorneys for Plaintiffs*
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JURY DEMAND

Pursuant to Fed. R. Civ. P. 38(b), Plaintiffs hereby demand a trial by a jury on all issues so triable.

Dated: September 25, 2019

PEPPER HAMILTON LLP

By: s/ Jeffrey M. Goldman

Jeffrey M. Goldman
M. Kelly Tillery (*Pro Hac Vice*
application to be filed)

Attorneys for Plaintiffs

EXHIBIT “A”



US010011804B2

(12) **United States Patent**
Mancosky

(10) **Patent No.:** **US 10,011,804 B2**
(45) **Date of Patent:** **Jul. 3, 2018**

(54) **METHOD OF EXTRACTING CBD, THC, AND OTHER COMPOUNDS FROM CANNABIS USING CONTROLLED CAVITATION**

(71) Applicant: **Hydro Dynamics, Inc., Rome, GA (US)**

(72) Inventor: **Douglas G. Mancosky, White, GA (US)**

(73) Assignee: **EcoXtraction, LLC, Lafayette, LA (US)**

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

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(21) Appl. No.: **15/240,450**

(22) Filed: **Aug. 18, 2016**

(65) **Prior Publication Data**

US 2017/0051231 A1 Feb. 23, 2017

Related U.S. Application Data

(60) Provisional application No. 62/208,179, filed on Aug. 21, 2015, provisional application No. 62/279,234, filed on Jan. 15, 2016.

(51) **Int. Cl.**

C11B 1/10 (2006.01)
C11B 1/14 (2006.01)
C11B 1/02 (2006.01)
C11B 1/04 (2006.01)
C07D 309/18 (2006.01)

(52) **U.S. Cl.**

CPC **C11B 1/10** (2013.01); **C07D 309/18** (2013.01); **C11B 1/02** (2013.01); **C11B 1/04** (2013.01); **C11B 1/106** (2013.01); **C11B 1/14** (2013.01)

(58) **Field of Classification Search**

CPC **C11B 1/10**; **C11B 1/14**; **C11B 1/02**; **C11B 1/106**; **C11B 1/04**; **C07D 309/18**
See application file for complete search history.

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Primary Examiner — John M Mauro

(74) *Attorney, Agent, or Firm* — Jones Walker LLP

(57) **ABSTRACT**

A method of extracting CBD, THC, desirable oils, and other compounds from the cannabis plant includes drying the plant, grinding or chopping the plant, mixing the resulting pieces of the plant with a fluid to form a mixture, circulating the mixture through a controlled cavitation reactor to liberate CBD, THC, desirable oils, and other compounds from the pieces, and separating the liberated CBD, THC, desirable oils, and other compounds from the fluid. The method may be used as an adjunct to or in combination with traditional extraction techniques such as leaching to increase yields.

11 Claims, 2 Drawing Sheets

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FIG. 1

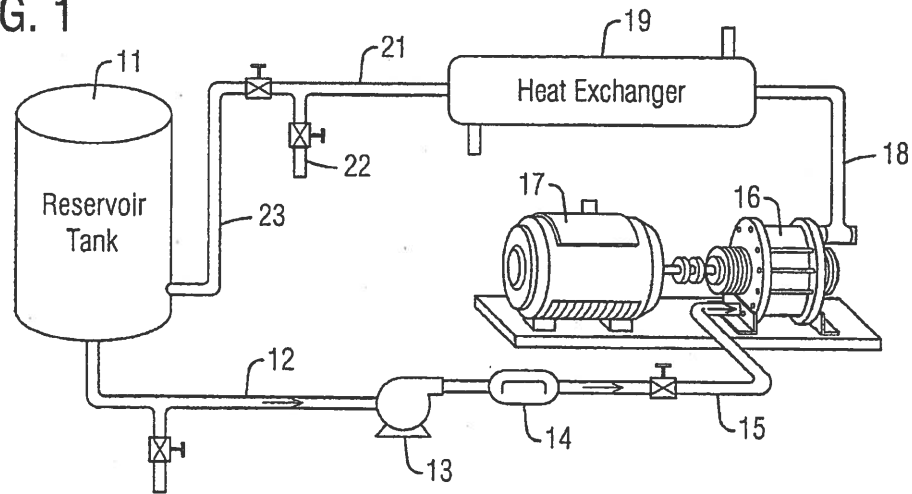
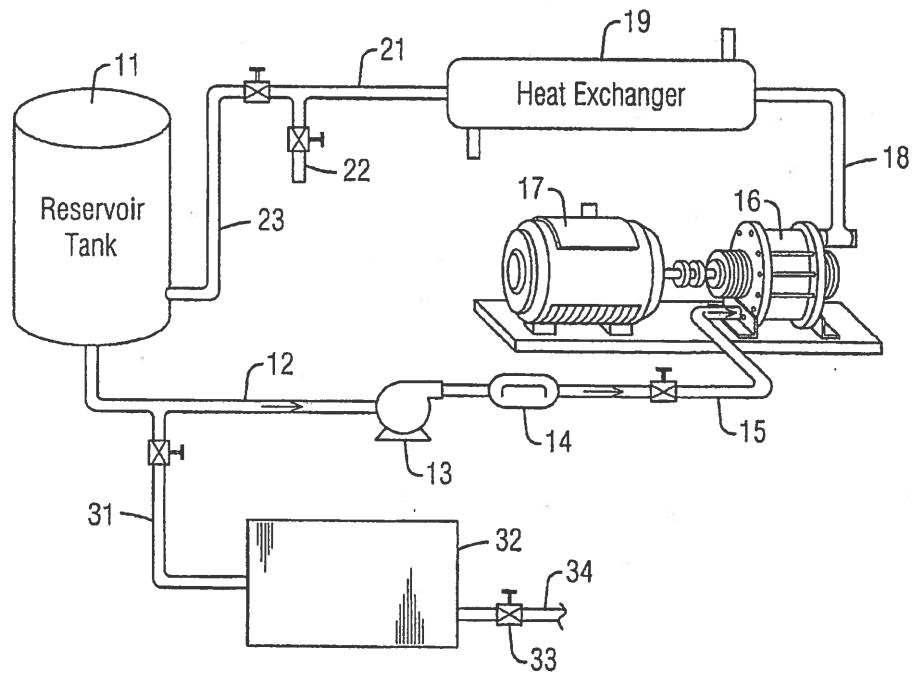


FIG. 2



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METHOD OF EXTRACTING CBD, THC, AND OTHER COMPOUNDS FROM CANNABIS USING CONTROLLED CAVITATION

REFERENCE TO RELATED APPLICATION

Priority is hereby claimed to the filing date of U.S. provisional patent application 62/208,179 filed on Aug. 21, 2015 and to the filing date of U.S. provisional patent application 62/279,234 filed on Jan. 15, 2016. The content of these two provisional patent applications is hereby incorporated by reference.

TECHNICAL FIELD

The invention exemplified herein with representative embodiments generally relates to extraction of compounds from biomass and more specifically to the extraction of cannabinoids (CBD), Tetrahydrocannabinol (THC), desirable oils, and other compounds from the cannabis plant.

BACKGROUND

Cannabis legalization in several jurisdictions has led to strong demand for products made from the oils and chemical compounds that can be extracted from Cannabis (referred to herein as "extracts") and used by individuals without smoking the plant itself. For example, demand has grown in the areas of Pharmaceuticals commonly referred to as "medical marijuana," and in nutritional supplements, edible products, and vaporizer fluids to name a few. Cannabis extracts are traditionally made by exposing cannabis plants to carbon dioxide, butane, propane, alcohol, glycerin, and/or other solvents in a vessel or mixer to leach the extracts from cannabis plants (a process referred to as leaching. Ultrasound also has been used in an attempt to liberate the extracts from the cannabis plant. These and other traditional techniques often suffer from issues such as low yield, high cost, safety issues, and/or contaminants that adversely affect the flavor or efficacy of the resulting extracts.

A need exists for a method of extracting CBD, THC, desirable oils, and other compounds from cannabis that addresses and resolves the problems and shortcomings of prior art leaching and other techniques, that is more efficient at extracting such compounds, that can be selective, and that does not introduce unwanted contaminants into the resulting extracts. There is a further need for a method and apparatus for maximizing the generally low extract yields that are characteristic of prior art extraction techniques. It is to the provision of such a method that the present invention is primarily directed.

SUMMARY

It has been shown that the pressure fluctuations induced by cavitation events can enhance extraction of diverse natural substrates, substances, and compounds from a range of lignocellulosic material including, for example, wood, corn, hops, and others. Certain of our issued patents and pending patent applications, including U.S. Pat. No. 8,430, 968, disclose various techniques for extracting compounds from lignocellulosic materials using controlled cavitation. Those patents and patent applications are hereby incorporated by reference. The inventors have discovered a method and apparatus for applying controlled cavitation technology to the extraction of CBD, THC, desirable oils, and other compounds from the cannabis plant. They also have discov-

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ered a method and apparatus for enhancing the effectiveness of such extraction methodology compared to the traditionally low yield prior art extraction techniques.

Briefly described, the method of the invention comprises drying the cannabis plant; optionally separating various parts of the plant such as leaves, stems, and seed; chopping or grinding the plant parts together or separately into small pieces; mixing the pieces with a fluid that may or may not contain solvents; and passing the mixture through a controlled cavitation zone. Within the cavitation zone, high intensity shock waves and the corresponding highly energetic pressure variations penetrate the cannabis pieces within the mixture. This, in turn, liberates CBD, THC, oils, and other compounds entrapped within the pieces through a number of processes. Such processes may include, for example, forcing fluid into and out of the pieces to mix with trapped oils and substances, lysis of hard-to-penetrate cells to liberate compounds trapped therein, and reversal of hornification due to drying. The result is higher yield, higher efficiency, and lower cost extraction. As an enhancement, the method and apparatus of the invention may be employed before or after traditional extraction techniques to increase the efficiency and yields produced by those techniques.

An apparatus for carrying out the method comprises a housing containing a rotor with cavitation inducing structures on its surface. A cavitation zone is defined between the surface having cavitation inducing structures and the wall of the housing. The rotor is rotated within the housing and the mixture of fluid and entrained cannabis pieces is pumped through the housing in such a way that the mixture passes through the cavitation zone. The cavitation inducing structures on the rotor cause highly energetic and continuous cavitation events within the fluid of the mixture, which induce high energy shock waves that travel through the mixture.

As a result of the shock waves, the cannabis plant pieces are exposed to extreme, rapid, and highly energetic pressure variations that force the fluid of the mixture into and out of the cannabis pieces. The cannabis pieces also are subjected to the high energies of the shock waves. This activity along with other processes such as lysis of cell walls caused by the energy of the shock waves, dislodges CBD, THC and other oils and compounds trapped in the lignin, trichrome, cells, and pours of the cannabis. These compounds become dissolved in the fluid of the mixture and can be separated from the fluid later through known separation techniques.

Accordingly, a method of extracting CBD, THC and other compounds from cannabis is now provided that is highly efficient, extracts more of the compounds than traditional extraction techniques, does so at a lower cost, can be controlled for selective extraction of particular compounds, and is readily scalable to virtually any commercial production rate. The method and apparatus also can be used to enhance traditional extraction techniques such as leaching to increase and maximize yields. These and other aspects, features, and advantages of the invention will be better understood upon review of the detailed description presented below taken in conjunction with the attached drawing figures, which are briefly described as follows.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is an illustration of one embodiment of a controlled cavitation apparatus usable for extracting CBD, THC, desirable oils, and other compounds from cannabis according to the methodology of the invention.

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FIG. 2 is an illustration of a controlled cavitation apparatus used as a pre-extractor in conjunction with a traditional extraction technique to enhance the efficiency and yield of the traditional extraction technique.

FIG. 3 is an illustration of a controlled cavitation apparatus used as a post-extractor in conjunction with a traditional extraction technique to enhance the efficiency and yield of the traditional extraction technique.

DETAILED DESCRIPTION

Reference will now be made in more detail to the accompanying drawing figures, wherein various reference numerals and labels identify the various parts of the apparatus. Referring to FIG. 1, an apparatus for carrying out the methodology of the invention includes, in one illustrative embodiment, a reservoir tank 11 for holding a mixture of a fluid and ground or chopped pieces of the cannabis plant. The fluid may be water, a solvent, a surfactant, a reactant, an acid, a base, combinations thereof, or any other fluid effective to liberate CBD, THC and other oils and products from the cannabis plant. Water is a preferred fluid. A pump 13 communicates with the reservoir tank 11 through a conduit 12 and is configured to pump the mixture from the reservoir tank 11, through a flow meter 14, through conduit 15, to and through controlled cavitation device 16.

The controlled cavitation device 16 includes a housing within which a rotor (not visible but disclosed in the incorporated references) that can be rotated at a preselected and controllable speed by motor 17. The rotor has cavitation inducing structures such as arrays of bores formed in its peripheral cylindrical surface. A cavitation zone having a predetermined dimension is defined between the peripheral surface of the rotor bearing the cavitation inducing structures and an interior surface of the housing. The flow path of mixture moving through the controlled cavitation device 16 moves the mixture through the cavitation zone.

Rotation of the rotor within the controlled cavitation device 16 as the mixture is pumped through the cavitation zone induces highly energetic cavitation events in the mixture. These cavitation events induce highly energetic shock waves and corresponding highly intense and rapid pressure fluctuations that propagate through the mixture within the cavitation zone. As detailed below, this environment liberates CBD, THC and other oils and compounds from the cannabis pieces within the mixture and these compounds become dissolved or entrained within the fluid of the mixture. Upon leaving the controlled cavitation device 16, the mixture flows through conduit 18, through a heat exchanger 19 for removing heat generated by the cavitation process, and through conduits 21 and 23 back to the reservoir tank 11. A sample line 22 may be provided for extracting samples of the treated fluid for testing.

The mixture may be circulated through the controlled cavitation device 16 numerous times until the extraction of CBD, THC and other oils and compounds is maximized. Alternatively, single pass treatment may well be acceptable in many instances. Subsequently, the remaining lignin from the cannabis pieces is separated from the mixture and the CBD, THC and other oils and compounds, now dissolved or entrained within the fluid of the mixture, can be separated from the fluid of the mixture through standard techniques. The efficiency yield of extraction in the present invention is substantially greater than prior art extraction techniques and methods, the cost is lower, and the present invention may be scaled up easily to virtually any commercial production rate. The invention can thus be used to extract desirable com-

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pounds such as CBD, THC and oils for medicinal or other legal uses from cannabis on a commercial scale.

In a preferred exemplary methodology, cannabis plant is first dried. After drying, the cannabis plant can be coarsely chopped or finely ground into relatively small pieces. Various elements of the plant such as the leaves, stems, and seeds can be separated for independent treatment, or the elements may be left in the mix and treated together. The ground plant pieces are mixed with water to form a mixture. Solvents other than water can be added to the mixture if desired, but this is not considered to be a requirement or limitation of the invention. Such solvents, if added, can include, for example, carbon dioxide, alcohols, glycerin, propane, and/or butane among others. In one embodiment, the preferred solvent is simply water, which eliminates the problem with prior art techniques wherein residual contaminants from harsher solvents may remain in the extracted product.

The mixture can be circulated through the apparatus at various rates and for varying periods of time, through varying circulation cycles, and with varying temperatures, solvents, and cavitation intensities to alter the amount and type of compounds extracted. For example, a more easily dislodged compound may be extracted using less energetic cavitation, leaving less easily dislodged compounds for later extraction with more energetic cavitation. The time that the mixture is exposed to cavitation can be varied from a few minutes to many hours to obtain the desired result. The heat exchanger 19 may or may not be incorporated into the circuit, but generally is desirable for longer circulation times to dissipate heat build-up caused by the energy imparted to the mixture by cavitation. Internal clearances within the controlled cavitation device 16 may be adjusted or selected to handle different size pieces of the cannabis plant entrained within the flow of the mixture. Surface area of the ground pieces is an independent variable.

The method of this invention offers many advantages over somewhat similar extraction technologies such as ultrasound. Ultrasound is typically difficult to scale-up and replicate laboratory results on a commercial scale. The use of controlled cavitation according to the present invention can provide a better result at nearly any commercial volume. Cavitation also produces more energetic shock waves than ultrasound in the sense that higher energy pressure variations are generated by the cavitation events. This may also result in faster and higher yield. Finally, ultrasound and other cavitation technologies rely on small clearances or high shear that are not conducive to extraction of compounds from natural substrates such as plant material. Extraction through controlled cavitation according to the present invention can easily incorporate plant and other lignocellulosic material because of its inherent low shear and relatively large clearances within the cavitation zone.

In addition to the cavitation induced pressure fluctuations forcing solvent into cannabis pieces to liberate an entrapped compound, these pressure fluctuations are also capable of lysis (breaking down the membranes) of pressurized bodies like cells. These cells often harbor desirable compounds that are otherwise difficult to liberate due to the resiliency of the cell membranes. The lysis coupled with cavitation provides increased yield and selectivity. Exposing cannabis pieces to cavitation according to the present invention also can reverse hornification. Hornification occurs when the pore structure of the dried cannabis plant dries and bonds to itself thereby limiting future extraction from the natural capillary system of the plant. The highly energetic cavitation induced pressure fluctuations and shock waves of the present inven-

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tion forces solvent into these dried structures and reopens them to near their original configuration before the plant was dried, again increasing yield.

The intensity of the cavitation and therefore the energy of the shock waves and pressure variations can be controlled by, for example, varying the rate of rotation of the rotor, so that a desired result is selectively achieved without causing other less desirable results. For example, many of the compounds of interest are contained in a plant structure called the trichome. By controlling the rotation rate of the rotor to obtain a predetermined cavitation intensity, the trichome can simply be knocked off the plant material for later extraction. A different cavitation intensity can be selected to burst the trichome to liberate the compounds therein. The intensity can be selected to burst the cells in the main body of the plant, which releases smaller amounts of desired compounds, but also may release some undesirable compounds. Multiple passes through the controlled cavitation device setting different cavitation energies each pass may be used to, for example, liberate compounds from plant structures that yield the purist compounds in initial passes while liberating compounds from harder-to-extract-from structures or structures that also yield undesirable compounds in later passes.

The invention also can be used as an enhancement to more traditional lower-yield extraction techniques to improve significantly the efficiency and yield of such techniques. In FIG. 2, a traditional extraction system 32, such as a leaching system for example, is configured to extract CBD, THC and other substances from cannabis in a traditional manner. Such systems are inherently inefficient in that much of the CBD, THC and other substances are not extracted and are left trapped in the plant material. The traditional system has an outlet 34 and a valve 33 for removing fluid that contains the extracted compounds when the treatment is complete. In this embodiment, the cavitation extraction system of FIG. 1 is arranged upstream of the traditional extraction system in a pre-treatment configuration. Water or solvent containing CBD, THC and perhaps other compounds extracted in the cavitation extraction system can be delivered to the traditional extraction system 32 through conduit 31 for additional treatment. The initial pre-treatment in the cavitation extractor may serve to open pores, penetrate cell walls, and open capillaries in the cannabis plant as it extracts some of the CBD, THC and other compounds. This, in turn, can enhance the efficiency of the traditional extraction process 32 by allowing the leaching solvent to penetrate the opened pores, breached cell walls, and opened capillaries generated in the pre-treatment process.

FIG. 3 illustrates a process in which the cannabis plant is initially treated in a traditional extraction system 36 and then delivered through conduit 37, valve 38, and pipe 39 to the cavitation extraction apparatus of FIG. 1. In this case, the cavitation extraction system is configured as a post-treatment system. After the traditional extraction system as extracted the CBD, THC and other compounds that it is capable of extracting, the resulting effluent is passed through the cavitation extractor to remove remaining CBD, THC and other compounds from the cannabis plant material. Since much of the CBD, THC and other compounds are extracted in the traditional extraction system 36, it should take less time to extract remaining CBD, THC and other compounds using the cavitation extraction technique of the present invention. The ultimate result is that the compounds become dissolved or entrained in the fluid and can be separated later through known techniques.

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The invention has been described above in terms and within the context of preferred embodiments and methodologies considered by the inventor to represent the best modes of carrying out the invention. It will be understood, however, that the invention certainly is not limited to the illustrated embodiments and methodologies. A wide range of additions, deletions, and modifications, both subtle and gross, might well be made to the illustrated embodiments by the skilled artisan without departing from the spirit of scope of the invention, which is delineated only by the claims. For example, while it is preferred to dry the cannabis plant and to chop the dried plant into small pieces, this is not necessarily a limitation of the invention. It is possible to carry out the methodology of the invention with undried cannabis plant material that is either chopped into pieces or left as a whole plant. The claims should not be construed to exclude carrying out the methodology with undried plant material.

What is claimed is:

1. A method of extracting oils from the cannabis plant comprising the steps of:

- (a) drying the cannabis plant;
 - (b) chopping or grinding the dried cannabis plant into pieces;
 - (c) combining the pieces of cannabis plant with a fluid to form a mixture;
 - (d) passing the mixture through a cavitation zone;
 - (e) causing cavitation events in the fluid that produce shock waves and pressure variations in the cavitation zone, the cavitation zone being defined between the outer peripheral surface of a rotor and an interior surface of a housing within which the rotor is rotatably mounted, the rotor having cavitation inducing structures on its outer peripheral surface, and wherein the step of causing cavitation events comprises rotating the rotor within the housing as the mixture passes through the cavitation zone;
 - (f) as a result of step (e), liberating oils from the pieces of the cannabis plant, the liberated oils becoming entrained within the fluid; and
 - (g) separating the oils from the fluid;
- where in step (e), the shock waves and pressure variations are controlled by varying the rotation rate of the rotor to reverse hornification caused by step (a).

2. The method of extracting oils from the cannabis plant as claimed in claim 1 wherein step (f) comprises liberating CBD from the pieces of the cannabis plant.

3. The method of extracting oils from the cannabis plant as claimed in claim 1 where in step (c), the fluid comprises water.

4. The method of extracting oils from the cannabis plant as claimed in claim 1 where in step (c), the fluid comprises a solvent.

5. The method of extracting oils from the cannabis plant as claimed in claim 4 wherein the solvent is selected from a group consisting essentially of carbon dioxide, alcohol, glycerin, propane, butane, and mixtures thereof.

6. The method of extracting oils from the cannabis plant as claimed in claim 5 wherein the solvent is mixed with water to form the fluid.

7. The method of extracting oils from the cannabis plant as claimed in claim 1 further comprising the step of subjecting the mixture to a non-cavitation based extraction process prior to step (d).

8. The method of extracting oils from the cannabis plant as claimed in claim 7 wherein the non-cavitation based extraction process comprises leaching.

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9. The method of extracting oils from the cannabis plant as claimed in claim 1 further comprising the step of subjecting the mixture to a non-cavitation based extraction process following step (e).

10. The method of extracting oils from the cannabis plant as claimed in claim 9 wherein the non-cavitation based extraction process comprises leaching.

11. The method of extracting oils from the cannabis plant as claimed in claim 1 where in step (e), the shock waves and pressure variations are controlled by adjusting the rotation rate of the rotor to break down membranes of pressurized bodies in the pieces of the cannabis plant to release oils from the pressurized bodies.

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EXHIBIT “B”



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(12) **United States Patent**
Mancosky

(10) **Patent No.:** **US 9,469,548 B2**
(45) **Date of Patent:** **Oct. 18, 2016**

(54) **CONTINUOUS HYDRODYNAMIC
CAVITATION CRYSTALLIZATION**

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(*) Notice: Subject to any disclaimer, the term of this
patent is extended or adjusted under 35
U.S.C. 154(b) by 0 days.

(21) Appl. No.: **15/046,941**

(22) Filed: **Feb. 18, 2016**

(65) **Prior Publication Data**

US 2016/0244334 A1 Aug. 25, 2016

Related U.S. Application Data

(60) Provisional application No. 62/118,941, filed on Feb.
20, 2015.

(51) **Int. Cl.**

B01D 9/02 (2006.01)

C01F 11/18 (2006.01)

(52) **U.S. Cl.**

CPC **C01F 11/181** (2013.01)

(58) **Field of Classification Search**

CPC **B01D 9/02; B01D 9/00; C13K 1/10;**
C30B 17/00

USPC **23/295 R, 301**
See application file for complete search history.

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(57) **ABSTRACT**

A device and process for crystallizing a compound using hydrodynamic cavitation comprises the steps of mixing at least one stream of a feed stock solution containing the compound to be crystallized. The feed stock solution is caused to flow through the cavitation zone of a rotor-stator type hydrodynamic cavitation device. Cavitation events and resulting shock waves generated in the cavitation zone cause nucleation and the production of seed crystals within the solution. Possible recirculation of the solution through the cavitation device promotes crystallization at the sites of the seed crystals resulting in increasing larger crystals. The resulting crystallized compound can be extracted from the solution and used in product manufacturing and industrial processes. The rotor-stator design allows for high flow rates, high applied energy and robust service with minimized wear.

16 Claims, 2 Drawing Sheets

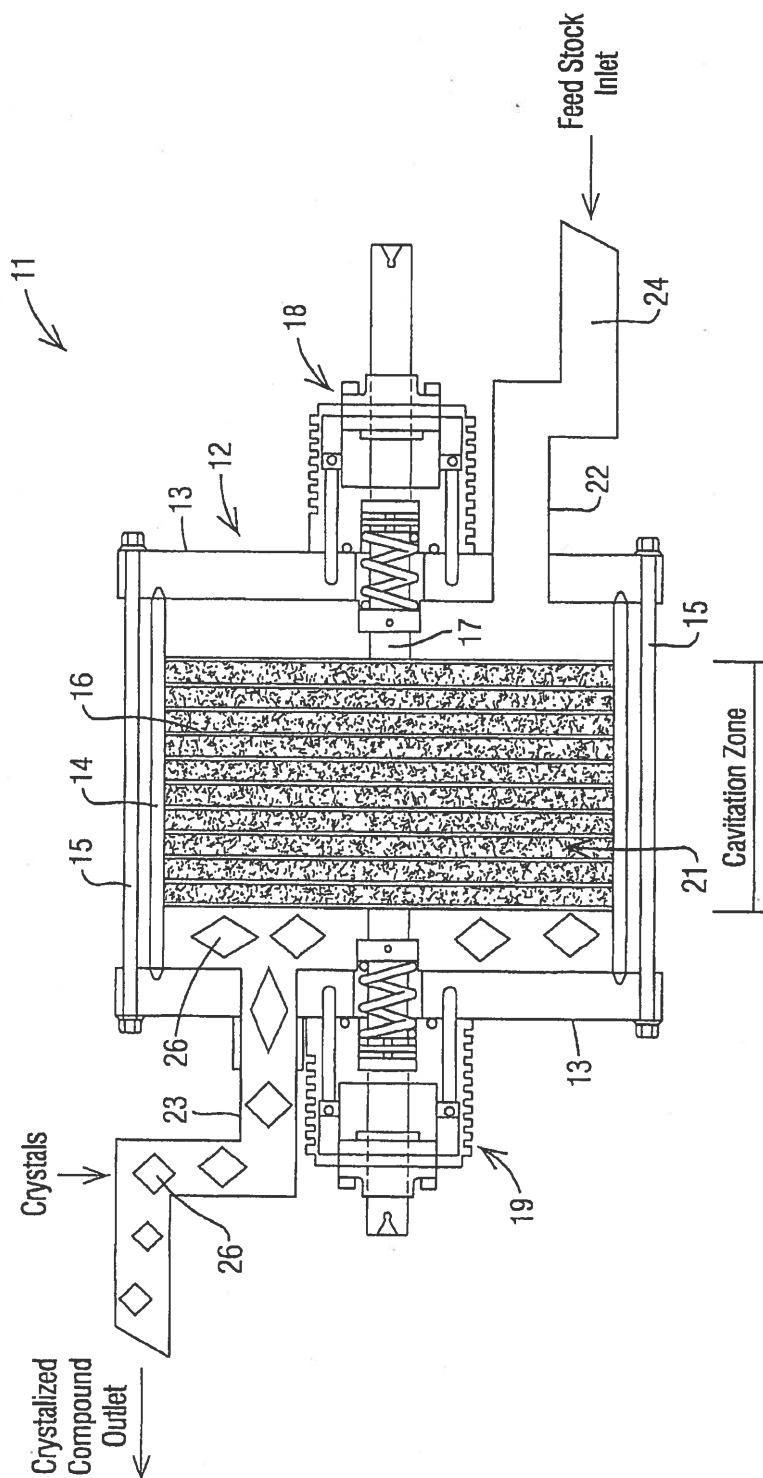
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FIG. 1



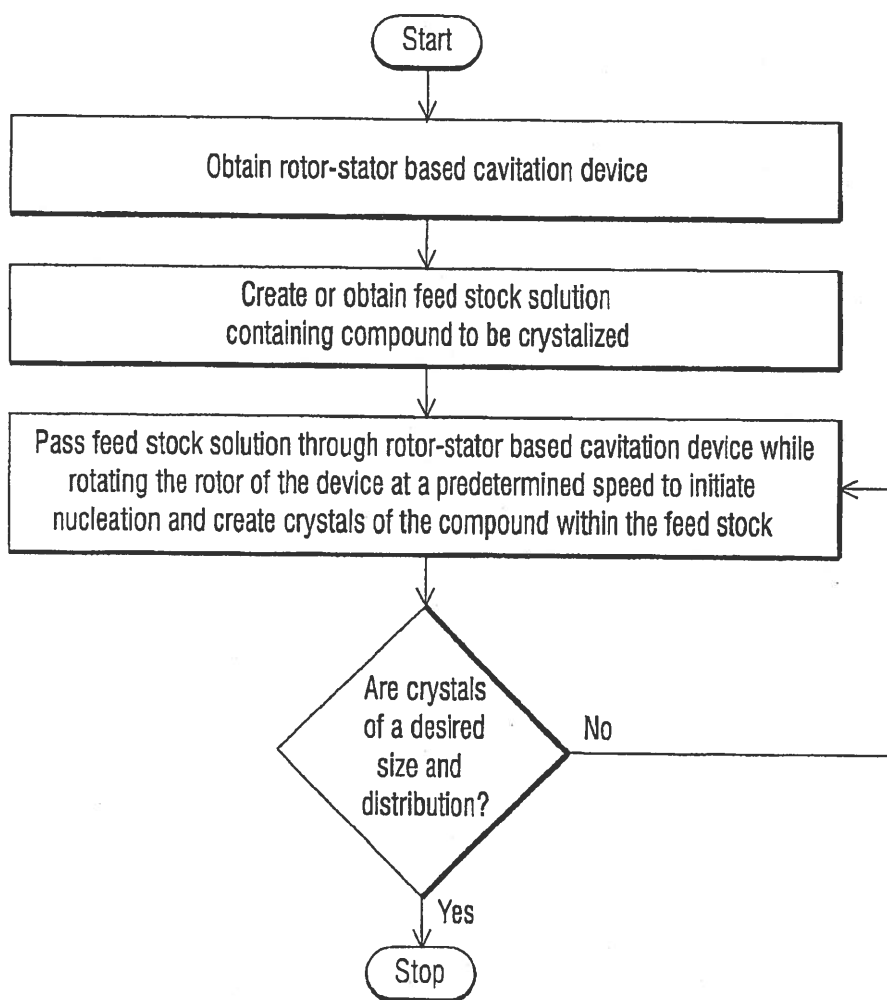
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FIG. 2



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CONTINUOUS HYDRODYNAMIC CAVITATION CRYSTALLIZATION

REFERENCE TO RELATED APPLICATION

Priority is hereby claimed to the filing date of U.S. provisional patent application 62/118,941 filed on Feb. 20, 2015, the entire contents of which is hereby incorporated by reference.

TECHNICAL FIELD

This disclosure relates generally to crystallization of compounds and more particularly to the creation of seed crystals in a compound to be crystallized by subjecting a flow of the compound at high rates to cavitation in a low shear environment.

BACKGROUND

Crystallization of compounds from solution is a separation and purification method used in the chemical, food, and pharmaceutical industries, particularly for the production of active compounds or their intermediates. Some of the goals of the crystallization process include producing a product meeting a desired purity level and also a product having the desired crystal size and size distribution. Crystallization from solution may be conducted as a batch process or as a continuous process. Batch crystallization equipment and operation is fairly simple but requires a significant investment of both time and money between batches. Additionally, batch crystallization suffers from quality control issues due to the lack of a steady state during the batch crystallization process. Continuous crystallization may be used for large volume commodity type materials with loose tolerances. This is because continuous crystallization does not typically permit adequate quality control to yield crystals suitable for use in industries demanding a high level of crystal size and size distribution control. U.S. Pat. No. 7,314,516 describes one apparatus and method that illustrates such a continuous crystallization process and the disclosure of this patent is hereby incorporated by reference.

One standard crystallization procedure involves contacting a supersaturated solution of a compound to be crystallized with an appropriate "anti-solvent" in a stirred vessel. Within the stirred vessel, the anti-solvent initiates primary nucleation which leads to crystal formation, sometimes with the help of seeding, and crystal digestion during an aging step. Mixing within the vessel can be achieved with a variety of agitators (e.g., Rushton or Pitched blade turbines, Inter-mig, etc.), and the process is done in a batchwise fashion.

Another known crystallization procedure for homogenous fluids employs temperature variation in a solution of a compound to be crystallized in order to generate a super saturation of the compound in the solution. Crystallization can then proceed from the super saturated solution.

It has been well known that ultrasound and cavitation can assist in nucleation of a compound within a fluid, although the mechanism is not completely agreed upon or understood. Subjecting a compound to be crystallized to cavitation energy is also known for generating nucleation sites and crystals of substantially uniform size. This is due, some think, to the high pressures and temperatures to which the compound is subjected in the immediate vicinity of the locations of cavitation bubble collapse.

A need exist for a continuous nucleation crystallization device and method that is capable of high throughput rates

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for extended periods of time yet that can produce nucleation and crystallization that is controllable and predictable. It is to the provision of such a device and method that the present invention is primarily directed.

SUMMARY

Briefly described, an apparatus and associated method are disclosed for creating nucleation and crystallization in a flow of feed stock solution. More specifically, an apparatus and method are disclosed for crystallizing a compound using hydrodynamic cavitation induced within a cavitation zone between a spinning rotor and the outer wall (or another wall) of a cylindrical cavity or stator within which the rotor spins. The method includes the steps of mixing at least one stream of a feed solution of such compound to be crystallized and passing the stream through the cavitation zone between the spinning rotor and outer cavity wall of a hydrodynamic cavitation device. The surface of the rotor is provided with a multitude of shallow radial bores (or other cavitation producing features) and the movement of these bores induces intense cavitation events in the solution within the bores. The energy and pressure of the cavitation events within the flow of feed stock solution causes nucleation and the production of seed crystals. Repeat cycling of the flow through the cavitation zone of the device can cause further crystallization of the compound within the flow.

The rotor-stator design of the cavitation device allows for high flow rates of the feed stock, the ability to subject the flow to very high energy from cavitation events, and robust service with minimized wear. Further, the device operates continuously at high flow rates making the apparatus and method of this invention suitable for crystallization of compounds within a feed stock solution at commercially desirable flow rates. Finally, the process can be tuned to provide highly reliable results to yield crystals suitable for use in industries demanding a high level of crystal size and size distribution control while still providing continuous high flow rates. The process is aided by the low shear environment of the cavitation zone, which tends to stabilize the forming nucleation seeds and crystals. These and other features, aspects, and advantages will become more apparent upon review of the detailed description set forth below taken in conjunction with the attached drawing figure, which is briefly described as follows.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a cut-away drawing showing an apparatus for generating nucleation and crystallization in a flow of feed stock solution according to one aspect of the invention.

FIG. 2 is a flowchart illustrating one embodiment of the methodology of the invention.

DETAILED DESCRIPTION

Embodiments of an apparatus for carrying out the methodology of the invention can take on various forms including those disclosed in U.S. Pat. Nos. 8,465,642; 8,430,968; 7,507,014; 7,360,755; 6,627,784; 5,957,122; and 5,385,298, all of which are owned by the assignee of the present invention and each of which is hereby incorporated by reference in its entirety. With the disclosures of these incorporated patents in mind, the method and apparatus of the present invention will be described generally with reference

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to the annexed drawing figures, wherein reference numerals indicate corresponding parts of the apparatus and steps of the method.

FIG. 1 shows in partially cutaway and simplified fashion one embodiment of an apparatus 11 for carrying out the methodology of the present invention. The apparatus 11 comprises, in this exemplary embodiment, a cylindrical housing 12 formed of opposed end plates 13 joined by a cylindrical outer wall 14, all held together with circumferential bolts 15. A cylindrical rotor 16 is disposed within the housing 12 and the rotor 16 is rotatably mounted on a shaft 17 that, in turn, is journaled within appropriate bearings 18 and 19. The shaft 17 is coupled to an electric motor (not shown) that, when activated, rotates the rotor within the housing at a relatively high rotation rate. As disclosed in detail in the incorporated patents, the rotor 16 is sized such that a cylindrical space is defined between the peripheral surface of the rotor and the inside surface of the cylindrical outer wall 14. This space is referred to herein as the cavitation zone 21.

A fluid inlet 22 communicates with the interior of the housing 12 on one side of the rotor 16 and a fluid outlet 23 communicates with the interior of the housing 12 on the opposite side of the rotor. Other inlet and outlet arrangements are possible. For example, inlets can be formed through the sides while outlets can be formed through the cylindrical outer wall, or vice versa. In one embodiment inlets and outlets are both formed through the cylindrical outer wall and preferably are oriented generally tangentially thereto. Such an embodiment, referred to as tangential inlets and outlets, can be beneficial, particularly where a fluid being treated is abrasive. In such a scenario, the tangential inlets and outlets reduce sharp turns within the fluid, which can result in erosion of surfaces within the apparatus.

A feed stock solution containing a compound to be crystallized may be fed into the housing through the inlet 22. The solution then moves to the periphery of the housing, travels through the cavitation zone 21, and exits the housing through outlet 23. The placement of the inlet and outlet is such that all of the fluid introduced through the inlet 22 must pass through the cavitation zone 21 before exiting the housing through the outlet 23. In a tangential inlet and outlet configuration, for instance, the inlet may be located between rotors of a split rotor cavitator and the outlets located outboard of each of the rotors. Other configurations are possible.

A plurality of holes or bores is formed through the outer surface of the rotor in the illustrated embodiment. Rotation of the rotor with a fluid present in the housing induces violent cavitation in the fluid within the bores of the rotor. More specifically, cavitation bubbles are continuously created in fluid within the bores and continuously collapse within the bores. The collapsing cavitation bubbles generate intense localized shockwaves that travel from the bores through the fluid within the cavitation zone. The collapsing cavitation bubbles also generate heat within the fluid by exciting the molecules of the fluid. The ultimate result is that fluid within the cavitation zone is heated by the cavitation and subjected to intense shockwaves that travel throughout the fluid flowing through the cavitation zone.

It has been found that subjecting a solution containing the compound to be crystallized to the high energy of the shockwaves within the solution efficiently and continuously generates small seed crystals of the compound suspended in the fluid, a process known as nucleation. Once seed crystals are generated, additional molecules of the compound can attach themselves to the seed crystals thereby forming larger

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and larger crystals. Due to the violent activity throughout the cavitation zone, the seed crystals become uniformly dispersed throughout the feed stock solution, a condition that is highly desirable especially in the manufacture of products sensitive to crystal size and distribution. Furthermore, the level of shear forces to which the crystals are subjected in the cavitation zone is very low as a result of the relatively large clearance between the outer surface of the rotor and the inner wall of the housing. This is important because subjecting newly formed crystals to shear can break apart the crystals thereby negating the advantageous crystallization that results from the process of this invention.

Once seed crystals are generated, the feed stock solution can be directed from the apparatus 11 to a process tank where the seed crystals grow into larger and larger crystals through molecular attachment. However, this is not considered the most efficient and controllable technique for growing the crystals or maintaining a uniform crystal distribution. More preferably, after a first pass through the apparatus 11 to generate seed crystals through nucleation, the feed stock solution containing the compound to be crystallized is cycled back through the apparatus 11 or a like apparatus located downstream. This both maintains a uniform distribution of seed crystals within the feed stock and promotes growth of crystals around the seed crystals by subjecting the feed stock again to the high energy environment within the cavitation zone.

The feed stock solution can be recycled through the apparatus 11 or successive like apparatuses until the crystals entrained within the feed stock solution flow are of a desired size for a particular application and are uniformly distributed throughout the feed stock solution. In this way, the crystal size and distribution can be carefully controlled to produce crystallization of the compound within the fluid with a precision that previously has required small batch processing, but on a large and continuous scale useful in commercial production.

FIG. 2 is a flowchart showing the steps of the present methodology in one preferred form thereof. First, a rotor-stator based cavitation device is obtained. One suitable device is the Shockwave Power Reactor (SPR) device available from Hydro Dynamics, Inc. of Rome, Ga. Next, an appropriate feed stock solution is created or obtained with the feed stock being in liquid form and containing the compound that is desired to be crystallized. The feed stock is passed through the rotor-stator based cavitation device while the rotor of the device is rotated at a predetermined rotation rate. This generates the aforementioned cavitation zone and the feed stock solution containing the compound moves through the cavitation zone as it passes through the rotor-stator based cavitation device. Within the cavitation zone, nucleation of the compound is induced in the feed stock to produce seed crystals and crystallization begins to take place at the sites of the seed crystals to grow larger crystals. The process gradually forms crystals of the compound within the feed stock solution. After passing through the rotor-stator based cavitation device, the feed stock is examined to determine whether the crystals that have been generated are of a desired size and distribution within the feed stock solution. If not, the feed stock solution may be passed again through the rotor-stator based cavitation device to induce further nucleation and crystal growth through crystallization. Once the crystals within the feed stock solution are of a desired size and distribution, the circulation can be discontinued and the crystals can be extracted and/or used for their intended purpose.

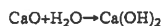
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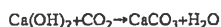
Sometimes nucleation and crystallization is desired in an abrasive mixture or with abrasives entrained within a flow. Petroleum waste water and inorganics in mining are common examples, but there are many others. Initiating nucleation and crystallization in such environments is difficult or impossible with traditional techniques. With the present invention, however, nucleation and crystallization is easily accomplished for compounds in abrasive fluids or fluids with entrained abrasives. The ability to accomplish this can be enhanced by replacing the side inlet and outlet shown in FIG. 1 of the apparatus with inlets and outlets that inject and withdraw fluid through the cylindrical wall of the housing in a direction tangential or substantially tangential to the direction of rotation of the rotor.

EXAMPLE

The production of Calcium Carbonate crystals was performed by mixing calcium oxide, water, and carbon dioxide to form a feed stock solution. The water is added to produce calcium hydroxide, and the carbon dioxide is introduced in this solution to precipitate the desired calcium carbonate, referred to in the industry as precipitated calcium carbonate (PCC). The calcium carbonate, water, and carbon dioxide solution was passed through a cavitation device such as that disclosed herein to form seed crystals through nucleation and the resulting solution was cycled again through a cavitation device to grow crystals of calcium carbonate around the seed crystals through crystallization. It was found that ultimate crystal size can be controlled quite precisely at high commercial flow rates by varying rotor-stator rpm and thus varying the amount of energy to which the feed stock is subjected within the cavitation zone. The reactions taking place within the cavitation zone were:



and



The resulting crystallization was found upon inspection to have produced crystals of uniform in size and uniform distribution throughout the solution.

The invention has been described herein in terms of preferred embodiments and methodologies considered by the inventor to represent the best modes of carrying out the invention. It will be understood by those of skill in the art, however, that a wide gamut of additions, deletions, and modifications, both subtle and gross, might well be made to the embodiments illustrated herein without departing from the spirit and scope of the invention. All such additions, deletions, and modifications are considered and should be construed to be within the scope of the invention exemplified in this disclosure.

What is claimed is:

1. A method of crystallizing a compound within a solution using controlled hydrodynamic cavitation, the method comprising the steps of:

- obtaining a cavitation apparatus having an inlet, an outlet, and an internal cavitation zone;
- causing the solution to flow through the cavitation apparatus in such a way that the solution moves through the cavitation zone;
- generating cavitation within the solution in the cavitation zone to induce nucleation of the compound to form seed crystals;

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distributing the seed crystals substantially uniformly throughout the solution;

promoting crystallization of the compound around the seed crystals to produce crystals within the solution; and

separating the crystals from the solution.

2. The method of claim 1, wherein the compound to be crystallized is an inorganic material.

3. The method of claim 1, wherein the compound to be crystallized is an organic material.

4. The method of claim 1 wherein the step of promoting crystallization of the compound comprises recycling the solution through the cavitation device.

5. The method of claim 1 further comprising the step of modifying the temperature of the solution within the cavitation device to control the rate of nucleation and cavitation.

6. The method of claim 1 wherein the step of promoting crystallization of the compound comprises moving the solution through a second cavitation device after seed crystals are formed in the solution.

7. The method of claim 1 wherein the compound comprises calcium carbonate.

8. The method of claim 1 further comprising the step of controlling the rate of flow of the solution through the cavitation device to control nucleation and crystallization.

9. The method of claim 1 wherein the step of distributing the seed crystals comprises subjecting the seed crystals to cavitation induced shock waves within the cavitation zone.

10. The method of claim 1 further comprising the step of adjusting the temperature of the solution prior to generating cavitation events in the solution to super saturate the solution with the compound.

11. The method of claim 10 wherein the step of adjusting the temperature comprises lowering the temperature.

12. A method of crystallizing calcium carbonate comprising the steps of:

mixing calcium oxide with water and carbon dioxide to form a feed stock solution;

moving the feed stock solution through a cavitation zone at a predetermined rate;

generating cavitation events within the solution in the cavitation zone, the cavitation events creating energetic shock waves that propagate through the solution;

the energetic shock waves promoting nucleation of calcium carbonate seed crystals within the solution;

distributing the seed crystals substantially uniformly throughout the solution;

promoting crystallization of calcium carbonate at the sites of the seed crystals to form calcium carbonate crystals; monitoring the size and distribution of crystals formed in the solution;

discontinuing the crystallization of calcium carbonate within the solution when the crystals formed in the solution reach a predetermined size and a predetermined distribution in the solution; and

extracting the calcium carbonate crystals from the solution for use.

13. The method of claim 12 wherein the step of distributing the seed crystals substantially uniformly throughout the solution comprises subjecting the seed crystals to cavitation induced shockwaves within the cavitation zone.

14. The method of claim 12 wherein the step of promoting crystallization of calcium carbonate comprises recirculating the solution and seed crystals through the cavitation zone.

15. The method of claim 12 further comprising reducing the temperature of the solution to supersaturate the solution.

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16. A method of inducing controllable nucleation and crystallization of a compound within a solution to produce crystals of the compound having a predetermined size range and a predetermined distribution throughout the solution, the method comprising the steps of:

- (a) establishing a treatment zone within a controlled cavitation reactor, the treatment zone having a dimension that results in relatively low shear when the solution is moved through the treatment zone at a predetermined rate;
- (b) passing the solution through the treatment zone at or below the predetermined rate to minimize shear within the solution;
- (c) inducing cavitation events in the solution within the treatment zone to cause energetic shockwaves to move through the solution to promote nucleation and consequent formation of seed crystals in the compound within the solution;
- (d) distributing the seed crystals substantially uniformly throughout the solution;
- (e) promoting crystallization within the solution wherein crystals of the compound grow larger at the sites of the seed crystals;
- (f) observing the size and distribution of the crystals within the solution;
- (g) discontinuing crystallization of the compound within the solution when the crystals reach a size within a predetermined range and a predetermined distribution throughout the solution.

* * * * *

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EXHIBIT “C”

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(12) **United States Patent**
Mancosky et al.

(10) **Patent No.:** **US 8,430,968 B2**
 (45) **Date of Patent:** **Apr. 30, 2013**

(54) **METHOD OF EXTRACTING STARCHES AND SUGAR FROM BIOLOGICAL MATERIAL USING CONTROLLED CAVITATION**

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(73) Assignee: **Hydro Dynamics, Inc.**, Rome, GA (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 701 days.

(21) Appl. No.: **12/357,465**

(22) Filed: **Jan. 22, 2009**

(65) **Prior Publication Data**
 US 2009/0186383 A1 Jul. 23, 2009

Related U.S. Application Data

(60) Provisional application No. 61/011,864, filed on Jan. 22, 2008.

(51) **Int. Cl.**
C08B 30/00 (2006.01)
D21C 1/02 (2006.01)

(52) **U.S. Cl.**
 USPC 127/34; 162/50
 (58) **Field of Classification Search** None
 See application file for complete search history.

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(74) *Attorney, Agent, or Firm* — Womble Carlyle Sandridge & Rice, LLP

(57) **ABSTRACT**

A method of extracting sugar, starch, and/or carbohydrates from feed material such as corn or corn stover is disclosed. The feed material is mixed with liquid and perhaps accelerants to form a mixture. The mixture is pumped through a controlled cavitation reactor, where it is exposed to shockwaves from cavitation events. The shockwaves open pores in the feed material and force liquid in and out of the pores to liberate trapped sugars and starches, which are dissolved in the liquid for subsequent removal.

21 Claims, 3 Drawing Sheets

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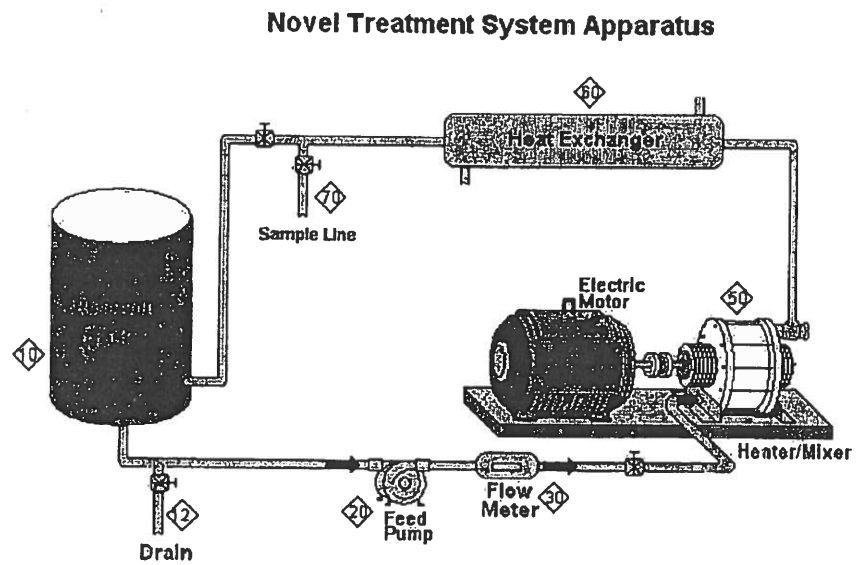


Figure 1

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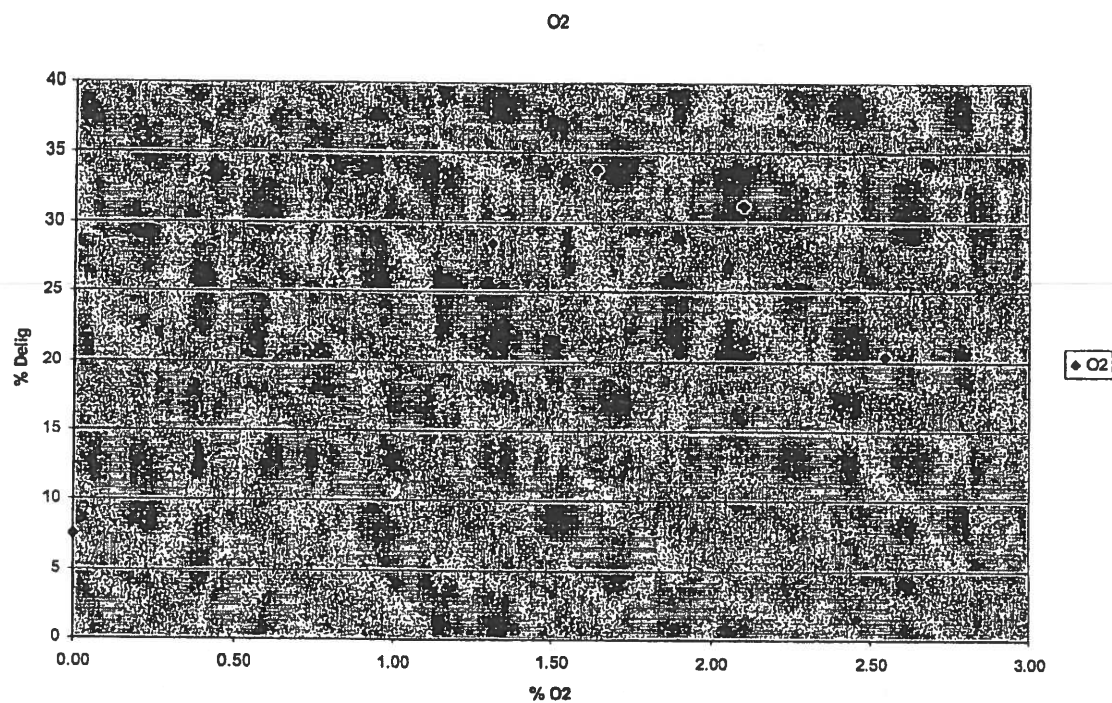


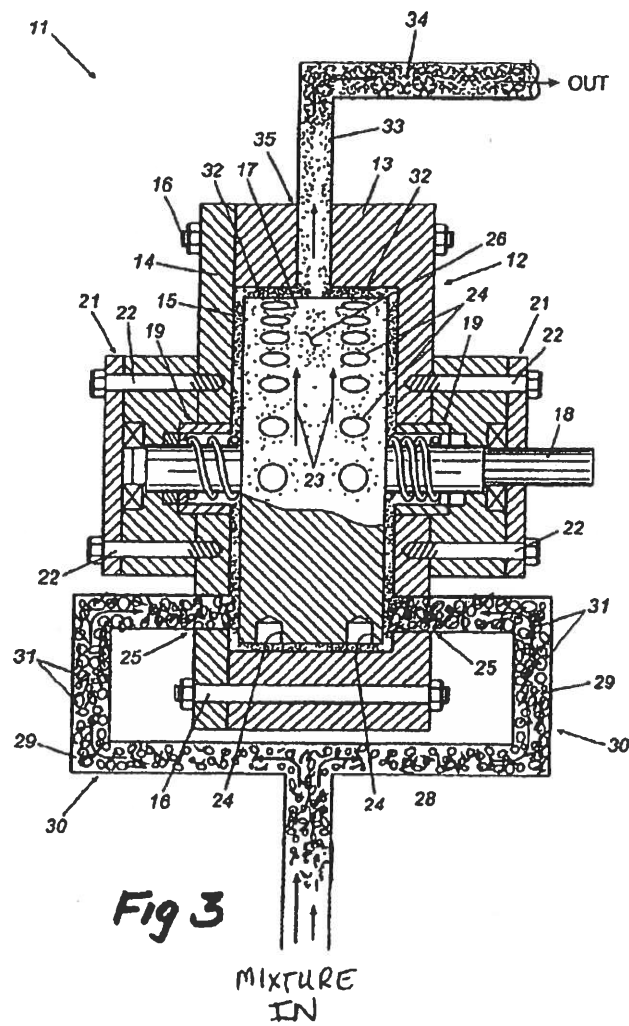
Figure 2

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METHOD OF EXTRACTING STARCHES AND SUGAR FROM BIOLOGICAL MATERIAL USING CONTROLLED CAVITATION

REFERENCE TO RELATED APPLICATION

Priority is hereby claimed to the filing date of U.S. provisional patent application Ser. No. 61/011,864 filed on Jan. 22, 2008, the disclosure of which is incorporated fully by reference.

TECHNICAL FIELD

This disclosure relates generally to the extraction of sugars and starches from biological material and more particularly to subjecting a mixture containing biological material to controlled cavitation to enhance such extraction.

BACKGROUND

In the ethanol industry, sugars and/or starches and/or carbohydrates are extracted from biological materials such as corn, grasses, switch grass, corn stover, or other lignocellulosic biomass containing sugars and/or starches. The extracted sugars and/or starches are then fermented to convert them to ethanol, and the mixture may be distilled to remove the ethanol. Historically, sugars and starches have been extracted by soaking biological materials, such as corn, in a mixture of water and perhaps a substance to help leach out the sugars and starches such as an acid, a caustic, enzymes, and the like. The more material that can be extracted or leached, the higher the yield for a given amount of biological material. There is a need for a more efficient method of extracting or leaching sugars and/or starches and/or carbohydrates from biological feed materials that produces higher yields. It is to the provision of such a method and an apparatus for carrying out the method that the present invention is primarily directed.

SUMMARY

Briefly described, a method of extracting sugars and/or starches and/or carbohydrates from biological feed materials is disclosed. The method comprises providing a controlled cavitation reactor such as that disclosed in U. S. Pat. No. 6,627,784, the disclosure of which is incorporated by reference. The reactor generally comprises a housing defining an internal cylindrical chamber and a cylindrical rotor rotatably mounted in the chamber. The rotor has a peripheral surface spaced from a cylindrical peripheral wall of the chamber to define a cavitation zone. A plurality of bores are formed in the peripheral surface of the rotor and these bores induce cavitation within them, which projects shockwaves into and through the cavitation zone when the rotor is rotated by an external motor. The method further comprises pumping through the cavitation zone a mixture of water and biological feed material containing sugars and/or starches and/or carbohydrates to be extracted. Catalysts or accelerants such as an acid, a caustic, and enzyme, or the like may be included in the mixture. While the mixture resides in the cavitation zone, the cavitation events occurring within the bores of the rotating rotor induce relatively large shockwaves and ultrasonic pressures within the mixture. This, in turn, opens the pores in the biological material and also moves liquids and accelerants within the mixture into and out of the pores. The trapped sugars, starches, and/or carbohydrates are thereby liberated and extracted from the biological material generally faster

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and more efficiently than with prior art soaking and mixing techniques. The reactor can be inserted in-line as a side stream of an already existing batch extraction process or can be the primary component of a continuous process unassociated with a traditional batch process. The reaction or extraction time per volume of mixture stays the same or is reduced. Therefore, an improved and more efficient method of extracting sugars, starches, and/or carbohydrates from biological feed material such as corn is disclosed. A better understanding of the method and an apparatus for carrying out the method will be obtained upon review of the detailed description set forth below when taken in conjunction with the accompanying drawing figures, which are briefly described as follows.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1. Illustrates one embodiment of an apparatus for carrying out the method disclosed herein.

FIG. 2 is a chart illustrating the results of a test of the method and apparatus of this disclosure.

FIG. 3 is a partially sectioned view of a controlled cavitation reactor for carrying out the method illustrating some of the interior components thereof.

DETAILED DESCRIPTION

A small volume cavitation reactor creates shockwaves that can open pores in biological material. This ability was demonstrated in oxygen delignification for the pulp and paper industry in which lignin was able to be effectively leached from the fibers without the use of oxygen. In conventional extraction, the raw material is heated in a reactor or vessel in the presence of a base at elevated temperatures and often is held in the vessel for hours or days. It has been discovered that, using a controlled cavitation reactor, the extraction of sugars, starches, and/or carbohydrates may be shortened to minutes and may produce higher extraction yields than with traditional batch extraction processes. This process may be especially useful for the extraction of sugars and starches from otherwise less productive products such as corn stover.

Referring to the drawing figures, FIG. 1 illustrates an apparatus for carrying out the extraction method of this invention. The apparatus includes a reservoir tank 10 for storing a mixture containing the sugar and starch bearing feed material, which may be grasses, corn, corn stover, or the like; water; and perhaps other materials designed to accelerate the extraction, such as an acid, a base, an enzyme, or combinations thereof. The mixture also may contain no accelerants and may just be a mixture of feed material and water. The mixture is drawn from the reservoir tank 10 by a feed pump 20 and pumped through a flow meter 30 and to a controlled cavitation reactor 50. Within the reactor 50, the mixture is exposed to cavitation and/or the resulting shockwaves and ultrasonic pressures. These shockwaves and pressures open the pores of the feed material and move liquids and any accelerants in the mixture into and out of the opened pores. This, in turn, liberates trapped sugars, starches, and/or carbohydrates from the feed material and these liberated substances become dissolved in the liquid within the mixture.

The mixture with dissolved liberated sugars and starches moves from the reactor 50 through a heat exchanger 60, where excess heat is removed from the mixture. The mixture then moves back to the reservoir tank 10. The contents of the reservoir tank 10 may be cycled through the reactor as described many times until a predetermined percentage of the sugars, starches, and/or carbohydrates has been removed

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from the feed material. Samples of the mixture may be removed through a sample line 70 and tested to determine when the maximum percentage has been removed. When the contents of the reservoir has been sufficiently treated, the reservoir, which now contains a liquid component rich in dissolved sugars, starches, and/or carbohydrates, may be removed through drain 12 for further processing into fuels such as ethanol.

The mixture itself may be from about 10 to about 50 wt % and more preferably from about 20 to about 30 wt % of feed material such as corn solids. Where accelerants or catalysts are used in the mixture, these substances may comprise from about 1 to about 12 wt % of the mixture. The remaining composition of the mixture may be water.

FIG. 2 is a graph showing the results of early tests of exposing lignocellulosic pulp to cavitation in an effort to remove lignin from the pulp. In these tests, a mixture of water, lignocellulosic pulp, and O₂ was exposed to cavitation within a controlled cavitation reactor. The test included one data point where no or 0% O₂ was added to the mixture. One would expect no lignin to be extracted under these circumstances. However, as is shown in the chart of FIG. 2, about 7 or 8 percent delignification occurred. Delignification is a multi-stage process. It was surmised that what had occurred to cause the observation was the removal of lignin that had been fragmented chemically in a previous stage, but that could not be removed by the standard agitated washing procedure followed by vacuum filtration. From these tests, it became clear that exposing the lignocellulosic material to cavitation and the resulting shockwave activity represented an improved and a more efficient method of removing lignin. It then occurred that, since corn and corn stover is somewhat similar to pulp fibers, a similar methodology might be transferred to the corn ethanol process to assist in getting enzymes into the corn or cellulosic ethanol structure and getting the chemically fragmented starches and sugars back out. The cavitation shockwave pressure fluctuations push and pull like a plunger on the pores of these natural substrates, which opens the pores and moves enzymes and other liquid materials in and out of the pores to liberate the trapped sugars, starches, and/or carbohydrates.

FIG. 3 is a partially cross sectioned view of a controlled cavitation reactor 11 similar but not identical to the reactor 50 of FIG. 1 and illustrating the process of the present disclosure. The reactor 11 comprises a housing 12 that defines a generally cylindrical interior chamber 15. The chamber 15 has a generally cylindrical peripheral wall and side walls. A cylindrical rotor 17 is mounted on a shaft 18 journaled in respective bearings 19 so that the rotor 17 is rotatable within the cylindrical chamber 15. The shaft 18 is driven by, for example, an electric motor (FIG. 1) to rotate or spin the rotor within the chamber. As can be seen in FIG. 3, the rotor 17 has a peripheral surface 26 that is spaced from the peripheral inner wall of the cylindrical chamber 15. This spacing defines a generally cylindrically-shaped zone that is referred to herein as the "cavitation zone." It should be understood, however, that the cavitation zone may or may not itself contain cavitation events. A plurality of bores 24 are formed in the peripheral surface of the rotor and are arranged in this illustration in two spaced rows extending around the circumference of the rotor.

Inlets 30 are provided into the cylindrical chamber 17 and an outlet 33 is provided out of the chamber. In this embodiment, the inlets are located on the side walls of the housing and the outlet is located on the peripheral wall of the housing. It will be understood that other arrangements of inlets and outlets may be substituted, such as the arrangement illustrated in FIG. 1. In any event, a mixture 31 of water, feed material

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such as corn or corn stover, and/or an accelerant such as an acid, a base, a caustic, an enzyme, or otherwise, is feed to the chamber through the inlets 30. The inlets and outlets are arranged to ensure that the mixture traverses the cavitation zone before it is extracted from the chamber through the outlet 33. As the rotor is rotated rapidly in direction 23, cavitation events are generated within the bores 24 of the rotor. These cavitation events, in turn, create a cascade of shockwaves that radiate outwardly from the bores into the cavitation zone and through the mixture therein. The shockwaves successively and repeatedly bombard the feed material within the mixture imparting an ultrasonic "push and pull" effect of the surrounding liquid on the feed material. This, in turn, opens up the pores of the feed material and at the same time moves liquid and enzymes or another accelerant, if present, into and out of the pores. Trapped sugars, starches, and/or carbohydrates are thus liberated efficiently from the feed material and become dissolved in the surrounding liquid, from which they can be removed by traditional processes. Once treated, the mixture is pumped out the outlet and may be returned additional times if desired to remove a maximum amount of sugar, starch, and or carbohydrate from the feed material.

The invention has been described in terms of preferred embodiments and methodologies considered to represent the best mode of carrying out the invention. It will be understood, however, that various additions, deletions, and modifications might be made to the illustrated embodiments without departing from the spirit and scope of the invention as set forth in the claims.

What is claimed is:

1. A method of releasing carbohydrates from lignocellulosic biomass comprising the steps of:

- (a) mixing the lignocellulosic biomass with water to form a mixture;
- (b) moving the mixture through a controlled cavitation reactor to expose the mixture to shockwaves resulting from cavitation events so that the carbohydrates are released from the lignocellulosic biomass into the mixture to form a mixture containing dissolved carbohydrates; and
- (c) extracting the mixture containing dissolved carbohydrates from the controlled cavitation reactor.

2. The method of claim 1, wherein the controlled cavitation reactor includes a housing defining a cylindrical chamber, a cylindrical rotor rotatably mounted in the chamber, bores in a peripheral surface of the rotor, and a cavitation zone defined between the peripheral surface of the rotor and an interior wall of the chamber, the moving step comprising passing the mixture through the cavitation zone as the rotor rotates.

3. The method of claim 1, wherein the carbohydrates comprise at least one of sugars and starches.

4. The method of claim 1, wherein the lignocellulosic biomass comprises at least one of corn and corn stover.

5. The method of claim 1, wherein the lignocellulosic biomass comprises grass.

6. The method of claim 1, wherein the mixture in (a) comprises from about 10 to about 50 wt % lignocellulosic biomass.

7. The method of claim 1, wherein the mixture in (a) comprises from about 20 to about 30 wt % lignocellulosic biomass.

8. A method of releasing carbohydrates from pores of a lignocellulosic biomass, the method comprising the steps of:

- (a) mixing the lignocellulosic biomass with water to form a mixture;

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(b) opening the pores of the lignocellulosic biomass, wherein opening the pores of the lignocellulosic biomass comprises moving the mixture through a cavitation zone of a controlled cavitation reactor to expose the mixture to shockwaves resulting from cavitation events, so that the carbohydrates are released into the mixture to form a mixture containing dissolved carbohydrates;

(c) moving the mixture containing dissolved carbohydrates out of the controlled cavitation reactor; and

(d) separating the carbohydrates from the mixture containing dissolved carbohydrates.

9. The method of claim 8 further comprising moving the mixture containing dissolved carbohydrates through the cavitation zone of the controlled cavitation reactor at least an additional time before separating the carbohydrates from the mixture containing dissolved carbohydrates.

10. The method of claim 8, wherein the carbohydrates comprise at least one of sugars and starches.

11. The method of claim 8, wherein the lignocellulosic biomass comprises at least one of corn and corn stover.

12. The method of claim 8, wherein the lignocellulosic biomass comprises grass.

13. The method of claim 8, wherein the mixture in (a) comprises from about 10 to about 50 wt % lignocellulosic biomass.

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14. The method of claim 8, wherein the mixture in (a) comprises from about 20 to about 30 wt % lignocellulosic biomass.

15. A method of extracting carbohydrates from lignocellulosic biomass and forming ethanol from the carbohydrates, the method comprising mixing the lignocellulosic biomass with water to form a mixture, exposing the mixture to shockwaves created by cavitation events to release the carbohydrates from the lignocellulosic biomass to form a mixture containing dissolved carbohydrates, extracting the carbohydrates from the mixture containing dissolved carbohydrates, and fermenting the extracted carbohydrates to form ethanol.

16. The method of claim 15, further comprising distilling the mixture containing ethanol to extract the ethanol.

17. The method of claim 15, wherein the lignocellulosic biomass comprises lignocellulosic biomass.

18. The method of claim 15, wherein the lignocellulosic biomass comprises at least one of corn and corn stover.

19. The method of claim 15, wherein the lignocellulosic biomass comprises grass.

20. The method of claim 15, wherein the mixture of lignocellulosic biomass and water comprises from about 10 to about 50 wt % lignocellulosic biomass.

21. The method of claim 15, wherein the mixture of lignocellulosic biomass and water comprises from about 20 to about 30 wt % lignocellulosic biomass.

* * * * *

EXHIBIT “D”



US007507014B1

(12) **United States Patent**
League et al.

(10) **Patent No.:** US 7,507,014 B1
(45) **Date of Patent:** Mar. 24, 2009

(54) **CONTROLLED CAVITATION DEVICE WITH EASY DISASSEMBLY AND CLEANING**

2005/0042129 A1 2/2005 Kazem

(75) **Inventors:** Richard D. League, Muskegon, MI (US); Derek E. Parker, Lyerly, GA (US)

(Continued)

(73) **Assignee:** Hydro Dynamics, Inc., Rome, GA (US)

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(*) **Notice:** Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 331 days.

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(21) **Appl. No.:** 11/462,880

(Continued)

(22) **Filed:** Aug. 7, 2006

Primary Examiner—Charles E Cooley

(74) *Attorney, Agent, or Firm*—Womble Carlyle Sandridge & Rice

Related U.S. Application Data

(60) **Provisional application No. 60/705,752, filed on Aug. 5, 2005.**

(57) **ABSTRACT**

(51) **Int. Cl.**
B01F 7/02 (2006.01)
B01F 7/12 (2006.01)
(52) **U.S. Cl.** 366/172.1; 366/305; 366/349
(58) **Field of Classification Search** 366/64–66, 366/96–99, 262–265, 279, 302, 305, 349, 366/168.1, 285–286, 171.1–172.2; 99/348; 29/238, 239, 240; 122/26; 126/247
See application file for complete search history.

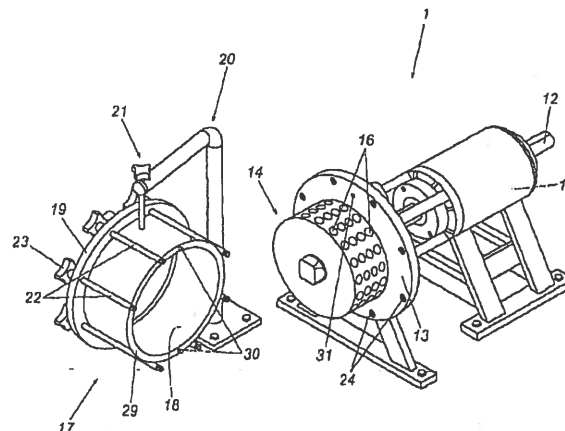
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A controlled cavitation device for mixing and heating of fluids in industries where sanitary conditions must be maintained. The device is easily disassembled for thorough cleaning and then easily assembled for continued operation. All internal surfaces are devoid of angles and are polished to food industry standards to maintain sanitary operation. The controlled cavitation device includes a cylindrical bearing housing for supporting a drive shaft that extends through each end of the bearing housing. An interior end plate is mounted to a C-face mount adjacent to the bearing housing and has a plurality of threaded bores around a periphery. A rotor is mounted adjacent to the interior end plate on the C-face mount. A housing assembly includes a cylindrical housing wall and is mounted on a swing arm assembly that is secured in place adjacent to the rotor. The housing assembly is rotated about a pivot arm in a first direction to fully enclose the rotor during assembly and is rotated in an opposite direction to expose the rotor during disassembly. An exterior end plate is fixed to one end of the housing wall to form a flange for connecting the housing assembly to the interior end plate.

23 Claims, 3 Drawing Sheets



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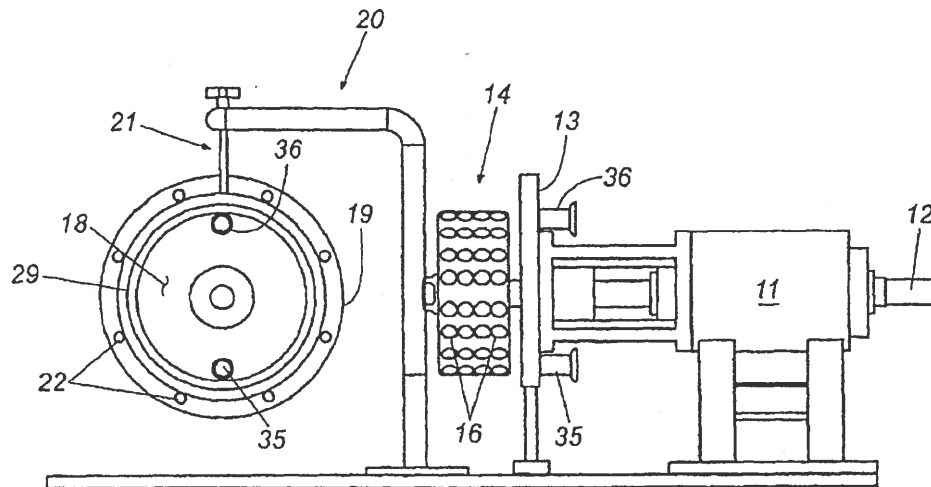


Fig. 3

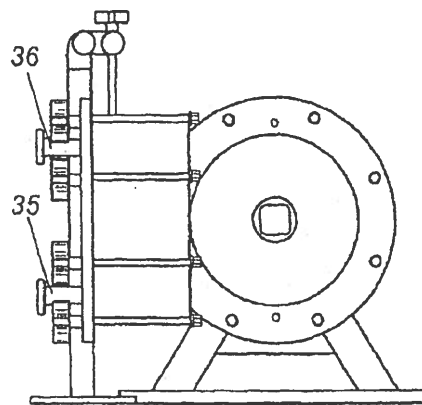


Fig. 4

U.S. Patent

Mar. 24, 2009

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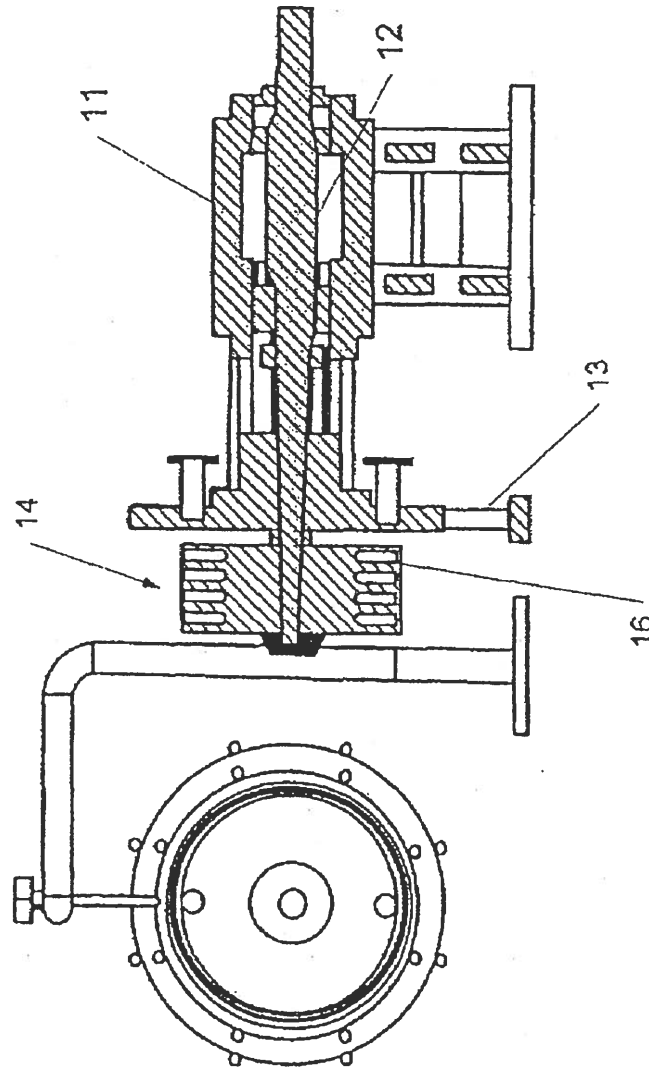


Fig. 5

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CONTROLLED CAVITATION DEVICE WITH EASY DISASSEMBLY AND CLEANING

CROSS-REFERENCE TO RELATED APPLICATION

The present patent application is a formalization of a previously filed, provisional patent application entitled "Controlled Cavitation Mixing Device with Easy Disassembly and Cleaning," filed on Aug. 5, 2005 as U.S. patent application Ser. No. 60/705,752, by the inventors named in this patent application. This patent application claims the benefit of the filing date of the cited provisional patent application according to the statutes and rules governing provisional patent applications, particularly 35 USC § 119 (e)(1) and 37 CFR §§ 1.78(a)(4) and (a)(5). The specification and drawings of the provisional patent application are specifically incorporated herein by reference.

FIELD OF THE INVENTION

The present invention relates generally to devices for heating fluids and mixing dissimilar fluids using controlled cavitation and more specifically to such controlled cavitation devices that can be disassembled, cleaned, and reassembled easily.

BACKGROUND OF THE INVENTION

The mixing and heating of fluids using controlled cavitation devices are known. The following U.S. patents and patent applications owned by the assignee of the present application teaches various aspects of a particularly successful controlled cavitation device for such purposes:

U.S. Pat. Nos. 5,188,090; 5,385,298; 5,957,122; 6,627,784
U.S. patent application Ser. Nos. 10/618,119; 10/843,104;
09/919,064; 10/932,604; 11/062,534; 60/654,387.

All of the disclosures and teachings of these patents and patent applications are hereby incorporated into this disclosure by reference as if fully set forth herein.

The devices disclosed in the aforementioned patents and patent applications have proven extremely efficient and useful for heating fluids and mixing dissimilar fluids. For certain heating and mixing applications, however, the devices disclosed have been found to exhibit certain shortcomings. For example, when heating and mixing fluids in the food processing industry, stringent sanitary and cleanliness requirements must be met. This means that the machinery used in the food processing industry (and other industries where stringent sanitary standards are enforced) must be able to be cleaned and thoroughly sanitized on a regular basis. Prior art controlled cavitation devices disclosed in the incorporated references can be disassembled, but such disassembly requires laborious, tedious, and time consuming efforts and generally requires special tools and an engineer or technician that is significantly skilled in the operation and maintenance of the device. This is not desirable for industries such as the food processing industry, which expect machines used for processing food to be able to be disassembled, cleaned, and reassembled quickly, easily, and by low-skilled food processing personnel. Thus, a need exists for a highly efficient controlled cavitation fluid heating and mixing device that meets this expectation so that its great efficiency can be exploited in industries such as food processing where easy and frequent cleaning of the device is required. It is to the provision of such a device that the present invention is primarily directed.

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SUMMARY OF THE INVENTION

Briefly described, the present invention, in one exemplary embodiment thereof, comprises a controlled cavitation device that can be disassembled, cleaned, and reassembled quickly and easily by relatively unskilled personnel. The configuration of the device is similar in many respects to the device disclosed in U.S. Pat. No. 5,957,122, which is incorporated by reference. Generally, the device includes a cylindrical rotor mounted on a shaft and disposed in a generally cylindrical housing. An electric motor or other motive means is coupled to the shaft (or the shaft can be an extension of the shaft of the motor itself). When the motor is activated, it spins the rotor at relatively high rotational speeds within its housing.

The rotor is provided on its outer periphery with a plurality of discontinuities, preferably in the form of an array of holes drilled into the rotor. Fluid to be heated or mixed is pumped through the space between the outer peripheral surface of the rotor and the cylindrical wall of the housing. As the rotor turns, violent cavitation events are induced in the fluid in the regions of the holes. The energy released by this cavitation heats the fluid and, where two fluids are introduced, has a wide variety of desirable effects including highly efficient mixing of the fluids, efficient inducement of chemical reactions within the fluids, and many others. Such effects are disclosed and discussed in detail in the patents and patent applications incorporated by reference.

In addition to the benefits of the devices and methods in the incorporated references, the present application provides structure and methodology for disassembling a controlled cavitation device easily for cleaning, and then easily reassembling the device for use. Generally, this is accomplished through a uniquely designed rotor housing assembly. The rotor housing generally is formed by a cylindrical peripheral wall that is capped on its ends by disc-shaped end plates. The end plates have a larger diameter than the wall so that a mounting flange is formed by the radially projecting edge portions of the end plates. The inboard end plate (i.e., the one closest to the motor) is mounted on and is a part of the support structure connected to the bearing housing. The rotor shaft extends through bearings in this end plate to be coupled through a bearing housing to the motor.

The outboard end plate is secured to the cylindrical wall. An array of bolts extends through the flange of the outboard end plate and is positioned therearound. The subassembly formed by the outboard end plate and the cylindrical wall is mounted to the inboard end plate to form the closed housing that encloses the rotor. Specifically, this subassembly is moved over the rotor and the bolts extending through its peripheral flange are threaded into threaded bores in the peripheral edge portion of the inner plate, thereby securely bolting the housing assembly together.

In the present invention, each bolt of the array of bolts used to secure the housing components together is provided with a hand knob on the outboard end plate in order to reduce the use of tools in the disassembly process, which is highly desired in the food processing industry. The subassembly comprising the outboard end plate and cylindrical wall of the housing is mounted on a swing arm located adjacent to the device. This allows the subassembly to be swung out of the way when disconnected so that it, and the then exposed rotor assembly, can be thoroughly cleaned. Afterwards, the swing arm allows the subassembly to be swung back into place easily and secured to the inboard end plate. Alignment pins or dowels are provided in the inside edge of the cylindrical wall and corresponding holes are provided in the inside plate. When the

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subassembly is properly aligned with the inside plate, the alignment pins register with the holes to position the subassembly properly before it is bolted in place. An O-ring in the edge of the cylindrical wall forms a seal against the inboard end plate to seal the interior of the housing.

BRIEF DESCRIPTION OF THE DRAWINGS

These and other advantages and aspects of the present invention will become apparent and more readily appreciated from the following detailed description of the invention taken in conjunction with the accompanying drawings, as follows.

FIG. 1 illustrates a perspective view of the controlled cavitation mixing device in an exemplary embodiment of the invention.

FIG. 2 illustrates a top view of the controlled cavitation mixing device in an exemplary embodiment.

FIG. 3 illustrates a side view of the controlled cavitation mixing device in an exemplary embodiment.

FIG. 4 illustrates an end view of the controlled cavitation mixing device in an exemplary embodiment looking back toward the rotor and motor assembly.

FIG. 5 illustrates a cross-sectional view of the exemplary controlled cavitation mixing device with the housing assembly in an open position.

DETAILED DESCRIPTION OF THE INVENTION

The following description of the invention is provided as an enabling teaching of the invention and its best, currently known embodiment. Those skilled in the art will recognize that many changes can be made to the embodiments described while still obtaining the beneficial results of the present invention. It will also be apparent that some of the desired benefits of the present invention can be obtained by selecting some of the features of the present invention without utilizing other features. Accordingly, those who work in the art will recognize that many modifications and adaptations of the invention are possible and may even be desirable in certain circumstances and are part of the present invention. Thus, the following description is provided as illustrative of the principles of the invention and not in limitation thereof since the scope of the present invention is defined by the claims.

Referring now in more detail to the drawing figures, wherein like reference numerals refer, where appropriate, to like parts throughout the several views, FIG. 1 illustrates most of the details of the controlled cavitation mixing device in an exemplary embodiment of the invention. As shown in FIG. 1, the mixing device 1 comprises a bearing housing 11 that rotatably supports a drive shaft 12. An inboard faceplate 13 is mounted to the bearing housing 11 with an appropriate bracket and the drive shaft extends through another bearing in the inboard faceplate to a free end portion. A cylindrical rotor 14 is secured to the free end portion of the drive shaft adjacent the inboard end plate and the rotor is formed with arrays of holes or bores 16 around its outer surface, as described in more detail in the incorporated references. Rotation of the drive shaft 12 by an electric motor (not shown) causes corresponding rotation of the rotor 14.

A housing subassembly 17 comprises a cylindrical housing wall 18 capped and sealed on one end by an outside or outboard end plate 19. The outboard end plate 19 (as well as the inboard end plate) is radially larger than the cylindrical wall 18, thus forming a peripheral flange 19 extending around the subassembly. An array of bolts 22 extend through holes in the flange 19 as shown and each bolt has an integral hand knob 23 by which it may be manually rotated. The flange formed by

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the inboard end plate 13 is provided with a corresponding array of threaded bores 24. When the subassembly 17 is moved over the rotor 14 and into contact with the inboard end plate 13, the bolts 22 are manually threaded by means of the hand knobs 23 into the threaded bores 24 to secure the housing subassembly 17 to the inboard end plate 13, thus forming a closed housing for the rotor.

A pair of alignment or locating pins or dowels 30 project axially from the end of the cylindrical wall 18 and these align with and extend into corresponding alignment holes 31 in the inboard end plate to ensure proper alignment before the bolts are tightened. Further, a flexible O-ring is embedded within an annular groove in the end of the housing subassembly and forms a secure seal with the inboard end plate when the two are bolted together. The housing subassembly is mounted on a custom swing arm assembly 20 that is secured in place adjacent to the rotor. More specifically, a rotating or pivoting pin 21 connects the subassembly 17 to the end of the pivot arm so that the housing subassembly is free to rotate about a vertical axis as well as be swung in an arc with the pivot arm. It will be appreciated that, with the forgoing configuration and arrangement of components, the housing subassembly can be disconnected easily and then swung conveniently out of the way on its swing arm assembly.

When the housing subassembly is disconnected and swung to the side, the rotor and housing interior surfaces are fully exposed so that they can be cleaned thoroughly, as is required in the food processing industry. To facilitate further the cleaning process, at least the interior stainless steel surfaces that contact food are polished to food grade specifications to inhibit growth of bacteria and the like and to prevent entrapment of food and contamination within discontinuities. Further, the holes in the rotor periphery surface, which actually create the cavitation events, are somewhat different than in the embodiments of the incorporated references. Specifically, the bottoms of the holes, rather than being flat or angled as in prior embodiments, are smoothly rounded and polished so that no abrupt intersections are formed. In fact, all internal angles of less than 135° have been eliminated in the present invention or have been formed with the proper radius as specified by 3-A sanitary standards for the purpose of eliminating nooks and crannies. Further, all internal surfaces are polished to an RA 32 (microinch) finish or better, according to the recommendations in ANSI/ASME B 46.1—Surface Texture. The elimination of internal angles and polishing of internal surfaces eliminates the buildup of contamination, encrusted food, and bacteria at such locations.

Referring to FIG. 2, hand knobs are also provided in place of traditional hexagonal bolts for securing and removing the inboard end plate to its mounting bracket. These hand knobs provide for easy disassembly for periodic and more thorough cleaning of the device where the rotor and inboard end plate are to be removed. This may occur, for instance, when the bearings in the end plate require cleaning or maintenance. As also shown in FIGS. 2 through 4, inlet ports 35 are provided near the bottom of each end plate and outlet ports 36 are provided near the top of each end plate. This symmetrical arrangement of inlet and outlet ports insures steady and balanced flow of fluid through the device and also eliminates unbalanced hydrodynamic pressures created by the inflow and outflow of fluid.

FIG. 5 illustrates a cross-sectional view of the controlled cavitation mixing device with the housing assembly in an open position. This view shows the rotor 14 secured to the bearing housing 11 adjacent end plate 13. The openings or holes 16 positioned on the outer surface of the rotor 14 are uniquely rounded at the bottom end to prevent contamination

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and entrapment of food items. The figure also depicts specially tapered bores for the shaft 12 and rotor 14.

The corresponding structures, materials, acts, and equivalents of all means plus function elements in any claims below are intended to include any structure, material, or acts for performing the function in combination with other claim elements as specifically claimed.

Those skilled in the art will appreciate that many modifications to the exemplary embodiment are possible without departing from the spirit and scope of the present invention for a controlled cavitation device that is easily disassembled and cleaned for use in sanitary applications such as in the food industry. In addition, it is possible to use some of the features of the present invention without the corresponding use of the other features. Accordingly, the foregoing description of the exemplary embodiment is provided for the purpose of illustrating the principles of the present invention and not in limitation thereof since the scope of the present invention is defined solely by the appended claims.

What is claimed:

1. A controlled cavitation device comprising:
a cylindrical bearing housing for supporting a drive shaft that extends through each end of the bearing housing;
an interior end plate mounted adjacent to the bearing housing and having a plurality of threaded bores around a periphery;
a rotor mounted adjacent to the interior end plate;
a housing assembly including a cylindrical housing wall mounted on a swing arm assembly including a pivot arm and secured in place adjacent to the rotor, wherein the housing assembly is rotated about the pivot arm in a first direction to fully enclose the rotor during assembly and is rotated in an opposite direction to expose the rotor during disassembly; and
an exterior end plate fixed to one end of the housing wall to form a flange for connecting the housing assembly to the interior end plate.
2. The controlled cavitation device of claim 1 wherein the exterior end plate has a plurality of openings for accepting a plurality of bolts to secure the housing assembly over the rotor to the interior end plate.
3. The controlled cavitation device of claim 2 further comprising a plurality of bolts, each bolt including an integral hand knob, the bolts extending through the plurality of openings in the exterior end plate and into a corresponding plurality of threaded bores in the interior end plate.
4. The controlled cavitation device of claim 3 further comprising an alignment pin extending axially from a surface on an interior end of the housing assembly for insertion into a corresponding opening in the interior end plate, the alignment pin facilitating an accurate alignment of the housing assembly when positioned over the rotor.
5. The controlled cavitation device of claim 4 further comprising a flexible O-ring that is embedded in an annular groove in the interior end of the housing assembly to form a seal with the interior end plate when the interior and exterior end plates are bolted together.
6. The controlled cavitation device of claim 1 further comprising a pivot pin to connect the housing assembly to an end of the pivot arm, the housing assembly being free to rotate about a vertical axis and to swing in an arc with the pivot arm.
7. The controlled cavitation device of claim 1 wherein the rotor comprises a plurality of shaped openings on an outer surface to generate a cavitation mixing of fluids during the mixing operation.

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8. The controlled cavitation device of claim 7 wherein the plurality of shaped openings are rounded on a bottom interior surface to avoid entrapment of mixed fluids during the mixing operation.

9. The controlled cavitation device of claim 7 wherein the plurality of shaped openings are rounded on a bottom interior surface to ensure all internal surface angles exceed 135 degrees.

10. The controlled cavitation device of claim 7 wherein all internal surfaces in the plurality of shaped openings are polished to meet food industry sanitary standards.

11. The controlled cavitation device of claim 1 further comprising an inlet port and an outlet port on both the interior and exterior end plates, each input port and outlet port located at diametrically opposite positions on each end plate to ensure a steady and balanced flow of fluids through the mixing device.

12. The controlled cavitation device of claim 1 further comprising a C-face mount attached to an end of the bearing housing for securing the interior end plate and rotor.

13. The controlled cavitation device of claim 12 wherein the interior end plate is secured to the C-face mount by bolts with integral hand knobs.

14. A controlled cavitation device comprising:
a bearing housing for supporting a drive shaft and having a C-face mount attached on one end;
an interior end plate secured to the C-face mount and having a plurality of threaded bores;
a rotor mounted adjacent to the interior end plate and secured to the C-face mount;
a housing assembly including a cylindrical housing wall mounted on a swing arm assembly including a pivot arm and secured in place adjacent to the rotor, wherein the housing assembly is rotated about the pivot arm in a first direction to fully enclose the rotor during assembly and is rotated in an opposite direction to expose the rotor during disassembly; and
an exterior end plate fixed to one end of the housing wall to form a flange for connecting the housing wall to the interior end plate.

15. The controlled cavitation device of claim 14 further comprising a plurality of bolts, each bolt including an integral hand knob and extending through an opening in the exterior end plate and into a corresponding threaded bore in the interior end plate.

16. The controlled cavitation device of claim 15 further comprising an alignment pin extending axially from a surface on an interior end of the housing assembly for insertion into a corresponding opening in the interior end plate, the alignment pin facilitating an accurate alignment of the housing assembly when positioned over the rotor.

17. The controlled cavitation device of claim 16 further comprising a flexible O-ring that is embedded in an annular groove in the interior end of the housing assembly to form a seal with the interior end plate when the interior and exterior end plates are bolted together.

18. The controlled cavitation device of claim 14 further comprising a pivot pin to connect the housing assembly to an end of the pivot arm, the housing assembly being free to rotate about a vertical axis and to swing in an arc with the pivot arm.

19. The controlled cavitation device of claim 14 wherein the rotor comprises a plurality of shaped openings on an outer surface to generate a cavitation mixing of fluids during the mixing operation.

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20. The controlled cavitation device of claim 19 wherein the plurality of shaped openings are rounded on a bottom interior surface to avoid entrapment of mixed fluids during the mixing operation.

21. The controlled cavitation device of claim 19 wherein the plurality of shaped openings are rounded on a bottom interior surface to ensure all internal surface angles exceed 135 degrees.

22. The controlled cavitation device of claim 14 further comprising an inlet port and an outlet port on both the interior

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and exterior end plates, each input port and outlet port located at diametrically opposite positions on each end plate to ensure a steady and balanced flow of fluids through the mixing device.

23. The controlled cavitation device of claim 14 wherein the interior end plate is secured to the C-face mount by bolts with integral hand knobs.

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EXHIBIT “E”



US006627784B2

(12) **United States Patent**
Hudson et al.

(10) Patent No.: **US 6,627,784 B2**
(45) Date of Patent: **Sep. 30, 2003**

(54) **HIGHLY EFFICIENT METHOD OF MIXING DISSIMILAR FLUIDS USING MECHANICALLY INDUCED CAVITATION**

(75) Inventors: Kelly Hudson, Gaylesville, AL (US);
Bijan Kazem, Woodstock, GA (US)

(73) Assignee: Hydro Dynamics, Inc., Rome, GA (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 198 days.

(21) Appl. No.: 09/747,469

(22) Filed: Dec. 20, 2000

(65) Prior Publication Data

US 2002/0077373 A1 Jun. 20, 2002

Related U.S. Application Data

(60) Provisional application No. 60/204,838, filed on May 17, 2000.

(51) Int. Cl.⁷ A62D 3/00; D21C 11/14

(52) U.S. Cl. 588/218; 516/10; 210/761;
162/29; 162/30.11; 422/185; 431/10

(58) Field of Search 516/10; 162/30.11,
162/30.1, 29, 31; 261/DIG. 71; 210/761;
422/185; 431/2, 10

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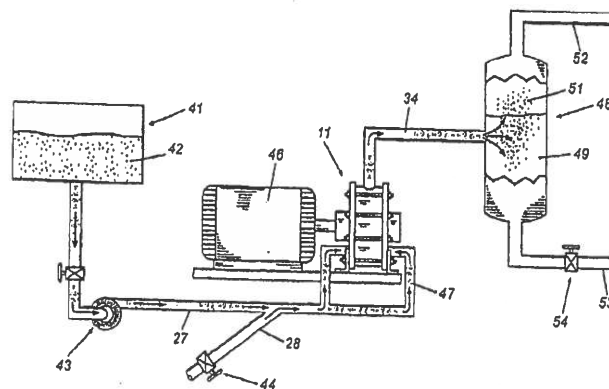
Primary Examiner—Daniel S. Metzmaier

(74) Attorney, Agent, or Firm—Womble Carlyle Sandridge & Rice, PLLC

(57) **ABSTRACT**

A method of mixing two or more dissimilar fluids such as a gas and a liquid is provided. The method includes the steps of introducing one fluid into the other to form a mixture, inducing cavitation in the mixture to reduce at least one of the fluids to a large number of relatively small units, and distributing the small units uniformly throughout the mixture. In a preferred embodiment, the step of inducing cavitation comprises introducing the mixture into a chamber having a rotating disk formed with a plurality of irregularities such as bores. The irregularities on the rotating disk induce cavitation in the mixture. The cavitation also breaks down van der Waals attractions within the mixture to enhance mixing.

5 Claims, 2 Drawing Sheets



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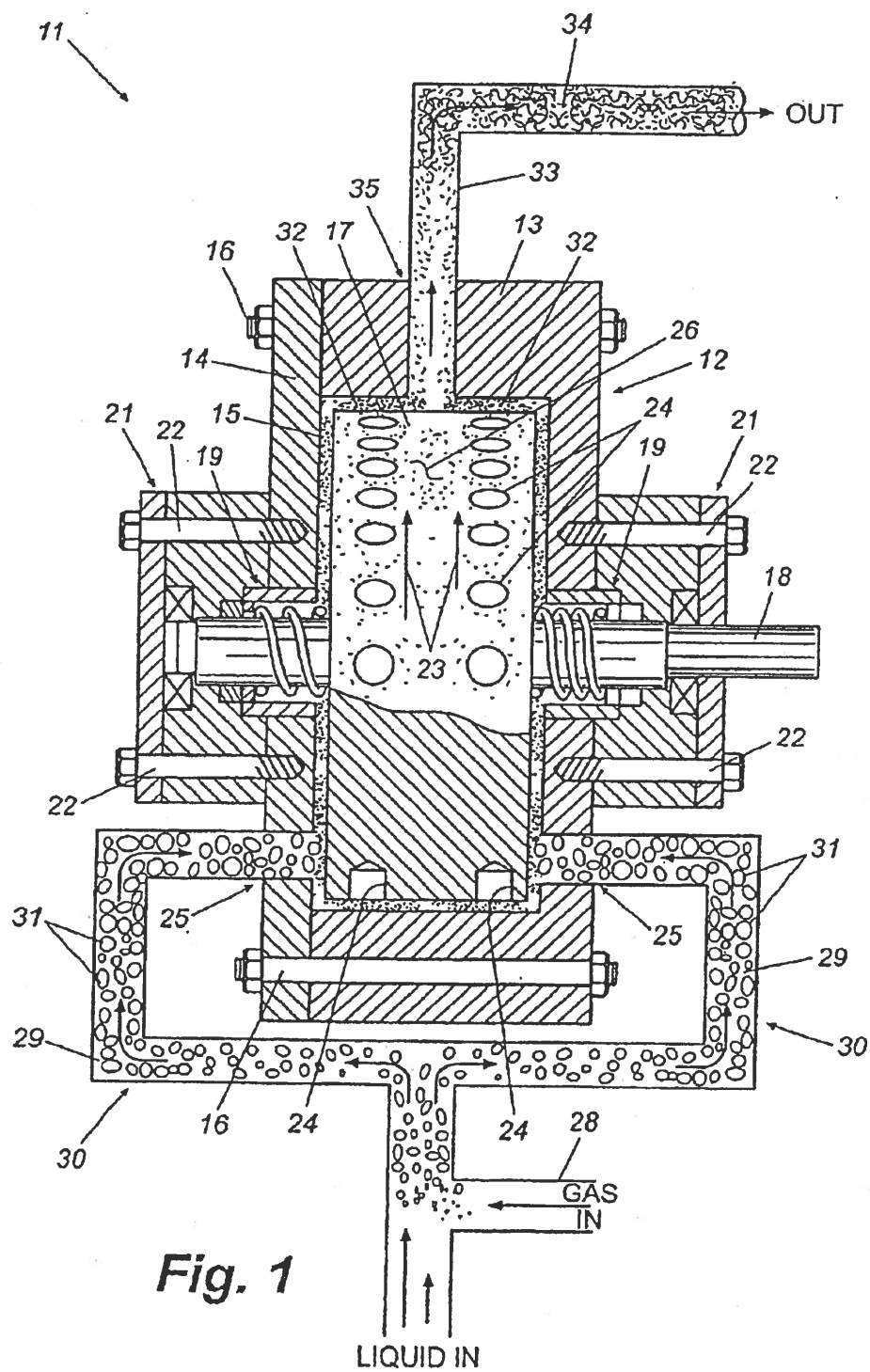
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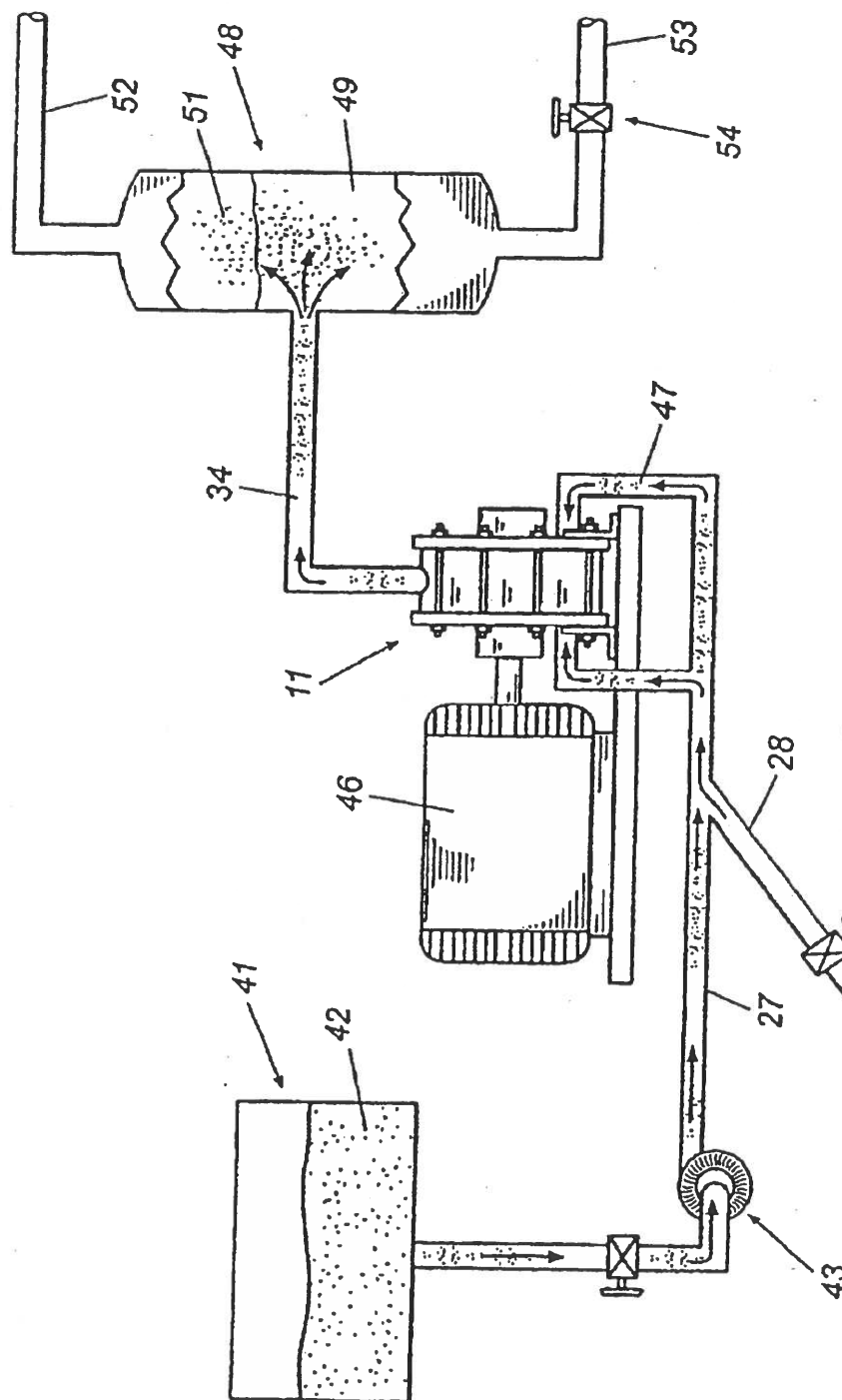


Fig. 2

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HIGHLY EFFICIENT METHOD OF MIXING DISSIMILAR FLUIDS USING MECHANICALLY INDUCED CAVITATION

REFERENCE TO RELATED APPLICATION

This application claims the benefit of the filing date of co-pending U.S. Provisional Patent Application Ser. No. 60/204,838 filed on May 17, 2000.

TECHNICAL FIELD

This invention relates generally to mixing and more specifically to methods and devices for mixing dissimilar fluids such as a gas and a liquid or two dissimilar liquids for industrial applications.

BACKGROUND OF THE INVENTION

The mixing together of dissimilar fluids such as a gas and a liquid or two dissimilar liquids has many industrial applications. For example, the mixing of oxygen and a liquid has applications such as the oxygenation of water for biological purposes and the oxygenation of fuel prior to burning to enhance combustion efficiency. The mixing of air and a liquid may be used in the pulp and paper, textile, and other industries in a process known as dissolved air floatation to separate suspended particulate materials from the liquid. Mixing industrial stack gasses with a liquid such as water is useful for removing environmental contaminants from the stack gasses prior to its release to the atmosphere. The mixing of dissimilar fluids such as, for example, oil and water, has industrial application in the creation of emulsions. Further, an existing emulsion may be separated into its constituent components by mixing it with another gas or fluid such as, for example, methane, which acts as an inhibitor to prevent recombination of the constituent components once they are broken apart.

One particular industrial application of gas/liquid mixing occurs in the pulp and paper industry where black liquor, which is a byproduct of cooking wood chips, often is recycled by being burned as a fuel in boilers. Even though such recycling is economically efficient, an emissions problem arises from the fact that untreated black liquor contains concentrations of Sodium Sulfide (Na_2S) that can be as high as 40 grams per liter or more. As a result, when untreated black liquor is burned, the Sodium Sulfide contained therein is converted to Sodium Dioxide (SO_2) and Hydrogen Sulfide (H_2S), which are known as totally reduced sulfur (TRS) compounds. TRS compounds are extremely harmful to the environment and therefore are highly regulated and may not be released to the atmosphere as a component of boiler stack gasses. Accordingly, black liquor often is treated before being burned in order to reduce or eliminate TRS emissions.

One method of treating black liquor prior to burning has been to mix or agitate it with air in a gas/liquid mixing process. When so mixed, Sodium Sulfide within the black liquor is oxidized in a chemical oxidation/reduction or "redox" reaction with oxygen molecules in the air and thereby converted to Sodium Thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$). Unlike Sodium Sulfide, Sodium Thiosulfate exists in a stable chemical state and thus does not participate in chemical reactions when the treated black liquor is burned in a boiler. Instead, the Sodium Thiosulfate simply precipitates to the bottom of the boiler, where it is ejected as a smelt.

Prior art industrial methods of mixing gases and liquids in general, and air and black liquor in particular, have involved introducing air in the form of bubbles into black liquor and

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agitating the mixture to break up the air bubbles and distribute them throughout the liquor. The goal, of course, is that oxygen molecules in the air will react chemically with or "oxidize" sodium sulfide molecules in the black liquor, rendering them inert during combustion of the black liquor. In one prior art process, such mixing is accomplished with rotating mechanical beaters having blades that impact and cut up the air bubbles while agitating the liquid. The problem with such a system, however, is that there is a natural lower limit to the size of the resulting air bubbles because larger bubbles cannot be cut or chopped to a size smaller than the size of the beater blades. Thus, the total composite surface area of the air bubbles in contact with the black liquor is severely limited. As a result, the probability that an oxygen molecule within an air bubble will come into contact with and oxidize a Sodium Sulfide molecule within the black liquor is reduced.

A further exacerbating problem and limitation of prior art gas/liquid mixing methods in general, and black liquor oxidation processes in particular, arises from the fact that the bubbles that are created by the mechanical beater blades of the mixing apparatus tend not to be distributed evenly throughout the black liquor. Instead, the bubbles, partially because of their relatively large size and partially because of the mechanical nature of the process, tend to agglomerate or concentrate into pockets of bubbles separated by relative voids in the liquor. This further reduces the probability that an oxygen molecule within an air bubble will come into contact with or "find" a Sodium Sulfide molecule and thus reduces the efficiency of the oxidation process. To address this inefficiency, it may be necessary to inject many times the amount of air necessary to oxidize the Sodium Sulfide into the mixer and to increase mixing times substantially to increase the probability of oxidation. However, such a brute force method of increasing oxidation efficiency substantially increases the time, energy, and resources required in the mixing process and thus introduces its own inefficiencies.

A final limitation of prior art gas/liquid mixing methods as applied to the oxidation of black liquor is imposed by the fact that the molecules within the liquor are attracted to each other by weak molecular forces known as van der Waals attraction. This results in the molecules clumping together in mutually attracted groups. In many cases, a Sodium Sulfide molecule that needs to come into contact with an oxygen molecule within a bubble in order to be oxidized may be surrounded within such a group by other molecules within the liquid and thus shielded from contact with a bubble and an oxygen molecule. In these cases, oxidation of the Sodium Sulfide molecule can not occur regardless of the volume of gas introduced or the length of the mixing process. This is due, in part at least, to the fact that the energy imparted to the liquor by mechanical beater blades is far less than that required to break the van der Waals attractions and free trapped molecules. In effect, then, the molecular van der Waals attraction within the liquor imposes a physical limit to the percentage of Sodium Sulfide molecules within black liquor that can be oxidized with traditional gas/liquid mixing techniques.

Thus, a specific need exists for a gas/liquid mixing method and apparatus applicable to the oxidation of black liquor in the pulp and paper industry that overcomes the problems, shortcomings, and limitation of prior art processes. More generally, a need exists for a new and unique method of mixing dissimilar fluids, be they gasses and liquids, dissimilar liquids, or otherwise, that is highly efficient, that results in virtually complete mixing in a short time and with a minimum of required energy and resources,

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and, in the case of oxidation applications, overcomes the physical limits on oxidation efficiency imposed by molecular van der Waals attraction. It is to the provision of such a method and an apparatus for carrying out the method that the present invention is primarily directed.

For clarity of disclosure and discussion, the present invention will be discussed herein primarily in the context of its application to the oxidation of environmental contaminants such as Sodium Sulfide in black liquor within the pulp and paper industry. Such an application is considered by the inventors to be a best mode of carrying out the invention. It will be understood and appreciated, however, that the method and apparatus of the invention is applicable to virtually any situation where dissimilar fluids are to be mixed together for industrial or commercial purposes. For example, the invention is applicable in the pulp and paper industry alone to a variety of processes including micro-mixing prior to gasification, mixing stack gases with black liquor in direct contact evaporators, mixing salt cake and black liquor, pulp drying, sludge dewatering, oxygen de-lignification, pulp bleaching by mixing pulp with ozone or other appropriate gases, and atomization of black liquor prior to its use in a recovery boiler. In the petroleum industry, the invention is applicable among other things to the separation of tight emulsions using micro-mixing and to heavy oil upgrading. Within the food processing industry, the mixing methodology of the invention has application in homogenization, oxygenation, and spice mixing processes. Applications within the environmental industry include oxidation/reduction of liquids or components of liquids, concentration and evaporation, BOD and COD reduction, dissolved air floatation, and fuel aeration. Thus, the discussion of the invention herein within the context of black liquor oxidation should not be interpreted as a limitation of the invention but only as representing a preferred embodiment or application and a best mode of carrying out the invention.

SUMMARY OF THE INVENTION

Briefly described, the present invention in a preferred embodiment thereof, comprises a unique and highly efficient method of mixing dissimilar fluids together by mechanically inducing cavitation within the fluids in a controlled manner. The result and goal is to obtain mixing on a microscopic level, uniform distribution of one fluid throughout another, and a breaking of van der Waals attractions between molecules within the fluids. In the preferred embodiment and best mode, the invention comprises a method of oxidizing environmentally hazardous compounds such as Sodium Sulfide within black liquor in the pulp and paper industry by mixing air with black liquor using controlled mechanically induced cavitation. The result is a virtually complete oxidation of the hazardous compounds and thus assurance that environmental toxins are not created when the black liquor is burned.

The methodology of the invention, in the context of oxidizing black liquor, comprises the steps of introducing and entraining air in the form of bubbles into a stream of black liquor to form a mixture of black liquor and air bubbles. The liquor/air bubble mixture is then directed into a hydrosonic mixer, which generally comprises a rapidly spinning rotor disposed within a cylindrical chamber within a housing. The rotor is provided with one or more arrays of relatively shallow holes or bores formed around its periphery. A space, referred to herein as a cavitation zone, is formed between the periphery of the rotor and the cylindrical wall of the housing chamber.

As the mixture of gas bubbles and black liquor passes through the cavitation zone, microscopic cavitation bubbles

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are continuously generated and collapse within the mixture by the action of the bores on the periphery of the spinning rotor.¹ The collapse of these cavitation bubbles creates violent and continuous cavitation within the gas/liquid mixture in the cavitation zone, and the energy of this cavitation acts to break up the air bubbles within the mixture into ever smaller bubbles or units. Since the minimum size of the air bubbles is not limited as in prior art mixers, the air bubbles are reduced by the cavitation into millions of substantially microscopic bubbles. Thus, the total surface area of air bubbles in contact with black liquor is significantly greater than in prior art mixers. The increased surface area increases the probability that an oxygen molecule within an air bubble will come into contact with and oxidize a Sodium Sulfide molecule within the black liquor. Further, because of the relatively violent agitation within the cavitation zone caused by rotor motion and cavitation effects, these microscopic air bubbles are mixed completely and uniformly throughout the black liquor, which further enhances the probability of contact between an oxygen molecule and a Sodium Sulfide molecule. Finally, the energy imparted to the mixture by the cavitation within the cavitation zone is more than sufficient to overcome the van der Waals attraction between molecules within the black liquor. This breaks apart the molecule clumps and frees Sodium Sulfide molecules that may be jacketed or shielded by other molecules within the black liquor. These freed molecules, then, are available to be contacted and oxidized by an oxygen molecule within one of the microscopic air bubbles.

The term "cavitation zone" is used herein to refer to the region between the outer periphery of the rotor wherein the bores are formed and the cylindrical wall of the housing chamber. This is where the most intense cavitation activity occurs. It should be understood, however, that cavitation may occur, albeit with less intensity, in regions other than this space such as, for example, in the reservoir or region between the sides or faces of the rotor and the housing. Thus "cavitation zone" is used herein to refer to the region of most intense cavitation, but should not be interpreted as an implication that cavitation cannot occur at some level in other regions of the hydrosonic mixer.

As a result of the creation and uniform distribution of microbubbles and the breaking of the van der Waals attractions, virtually complete oxidation of the Sodium Sulfide component of the black liquor is accomplished within the hydrosonic mixer. (Of course, a small amount of oxidation also may occur outside the mixer such as, for example, in the supply and outlet conduits of the system.) Further, the process can be accurately controlled by selecting rotation rate of the rotor and the amount of air initially introduced into the black liquor such that the complete oxidation is accomplished within a minimum time and with a minimum of energy and required introduction of air. The overall result is a gas/liquid mixing process that is far more efficient, faster, and more effective than is possible with prior art mechanical mixers. Once the Sodium Sulfide in the black liquor has been oxidized, the treated black liquor can be burned in a boiler with minimum of environmental toxins being produced and released to the atmosphere in stack gasses.

Thus, a method of mixing dissimilar fluids such as air and black liquor is now provided that addresses and overcomes the problems and shortcomings of the prior art. The method is highly efficient and effective and results in virtually complete oxidation of target components within black liquor or other liquids. These and other features, objects, and advantages of the methodology of this invention will become more apparent upon review of the detailed description set forth below when taken in conjunction with the accompanying drawing figures, which are briefly described as follows.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a partially sectioned view illustrating one preferred embodiment of an apparatus for mixing dissimilar fluids such as a gas and a liquid according to the present invention.

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FIG. 2 is an illustration of the apparatus of FIG. 1 functioning in a system for oxidizing black liquor for use in the pulp and paper industry.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

As mentioned above, the methodology of the present invention will be described within the context of its application to the oxidation of environmentally harmful compounds within black liquor in the pulp and paper industry. While this represents a preferred application and a best mode of carrying out the invention, it will be understood that the invention has numerous other applications wherever mixing of dissimilar fluids is required. In this regard, numerous examples of the application of the present invention in other mixing environments will be described following the detailed description of its application to black liquor oxidation.

Referring now in more detail to the drawings, in which like numerals refer to like parts throughout the several views, FIG. 1 illustrates a preferred embodiment of a hydrosonic mixer for carrying out the present invention. The hydrosonic mixer 11 comprises a cylindrical housing 12 defining an internal cylindrical chamber 15. In the illustrated embodiment, the housing 12 is formed of a casing 13 capped by an end plate 14 secured to the casing with bolts 16. Obviously, however, the housing 12 may be formed in other ways such as, for example, a central cylindrical shell capped by two end plates.

A cylindrical rotor 17 is disposed within the cylindrical chamber 15 of the housing and is mounted on an axially extending shaft 18. The shaft 18 is journaled on either side of the rotor within bearing assemblies 19 that, in turn, are mounted within bearing assembly housings 21. The bearing assembly housings 21 are secured to the housing 12 by means of appropriate fasteners such as bolts 22. The shaft 18 projects from one of the bearing housings 21 for being coupled to a motive means such as an electric motor 46 (FIG. 2). It will thus be seen that the rotor 17 may be spun or rotated within the cylindrical chamber 15 in the direction of arrows 23 by activating the motor 46 coupled to the shaft 18.

The rotor 17 has a peripheral surface that is formed with one or more circumferentially extending arrays of irregularities in the form of relatively shallow holes or bores 24. In the illustrated embodiment, the rotor 17 is provided with two arrays of bores 24 separated by a void 26, the purpose of which is described in more detail below. It should be understood, however, that fewer or more than two arrays of bores may be provided in the peripheral surface of the rotor as desired depending upon the intended application of the hydrosonic mixer 11. Further, irregularities other than holes or bores also may be provided. The rotor 17 is sized relative to the cylindrical chamber 15 in which it is housed to define a space, referred to herein as a cavitation zone 32, between the peripheral surface of the rotor and the cylindrical wall of the chamber 15.

Inlet ports 25 are provided in the housing 12 for supplying fluids to be mixed to the interior chamber 15 within the housing. Supply conduits 30 are coupled to the inlet ports 25. A liquid supply conduit 27 is coupled to the supply conduits 30 for supplying liquid such as black liquor to the hydrosonic mixer 11. A gas supply conduit 28 communicates with the liquid supply conduit 27 for introducing and entraining gas in the form of bubbles within the stream of liquid flowing through the liquid supply conduit 27.

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In the case of black liquor oxidation, within the context of which the present invention is described, black liquor is pumped through the liquid supply conduit 27 and air, which contains oxygen, is supplied through the gas supply conduit 28. At the junction of the liquid supply conduit 27 and the gas supply conduit 28, the black liquor and air form a gas/liquid mixture in the form of relatively large air bubbles 31 entrained within the flow of black liquor 29. This mixture of black liquor and air bubbles is directed into the cylindrical chamber 15 of the housing 12 through the supply conduits 30 and inlet ports 25 as shown. In the illustrated embodiment, an inlet port 25 is provided on either side of the housing 12 in order to equalize the hydraulic pressure on the rotor 17 to prevent undue stress on the bearing assemblies 19. However, this particular configuration is not a limitation of the invention and other configurations of inlet ports may be provided if desired.

An outlet port 35 is provided in the housing 12 and, in the illustrated embodiment, is located in the cylindrical wall of the housing to communicate with the cavitation zone 32 in a region of the rotor intermediate or between the arrays of bores 24. Location of the outlet port 35 in this way ensures that the entire volume of the gas/liquid mixture traverses at least one of the arrays of bores 24 and thus moves through a cavitation zone prior to exiting the hydrosonic mixer 11. Further, location of the outlet port 35 within the region of the inner chamber 15 aligned with the void 26 of the rotor prevents cavitation damage that otherwise might occur if the outlet port 35 were aligned with an array of bores 24. An outlet conduit 33 is coupled to the outlet port 35 for receiving treated black liquor and excess air from the hydrosonic mixer 11 and delivering it to a remote location for separation of the excess air from the treated black liquor and subsequent use of the treated black liquor.

In operation, the hydrosonic mixer 11 of FIG. 1 functions to oxidize environmentally harmful compounds within the black liquor essentially as follows. Black liquor, which contains, among many other compounds, the environmentally harmful compound sodium sulfide is pumped through the liquid supply conduit 27 to the supply conduit 30. Air is supplied through the gas supply conduit 28 to the stream of black liquor and the air and black liquor form a mixture comprised of relatively large air bubbles 31 entrained within the black liquor 29. The black liquor/air bubble mixture moves through the supply conduits 30 and enters the chamber 15 through supply ports 25 on either side of the rotor 17.

From the supply ports 25, the mixture moves toward the periphery of the rapidly rotating rotor 17 and enters the cavitation zones 32 in the region of the bores 24. As described in substantial detail in our previously issued U.S. Pat. No. 5,188,090, the disclosure of which is hereby incorporated by reference, within the cavitation zones 32, millions of microscopic cavitation bubbles are formed in the mixture within and around the rapidly moving bores 24 on the rotor. Since these cavitation bubbles are unstable, they collapse rapidly after their formation. As a result, the millions of microscopic cavitation bubbles continuously form and collapse within and around the bores 24 of the rotor, creating cavitation induced shock waves that propagate through the mixture in a violent albeit localized process.

As the mixture of black liquor and relatively large air bubbles moves into and through the cavitation zones 32, the air bubbles in the mixture are bombarded by the microscopic cavitation bubbles as they form and further are impacted by the cavitation shock waves created as the cavitation bubbles collapse. This results in a "chopping up" of the relatively large air bubbles into smaller air bubbles, which themselves

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are chopped up into even smaller air bubbles and so on in a process that occurs very quickly. Since the cavitation bubbles that cause the reduction of air bubbles into ever smaller air bubbles are microscopic in size, the practical lower limit to the size of the resulting air bubbles inherent in prior art mechanical mixing methods do not exist. Thus, the original air bubbles are continuously chopped up and reduced to millions of tiny microscopic air bubbles within the cavitation zone. The result is a total composite air bubble surface area in contact with the black liquor that is far greater than that possible in prior art mixing methods. Consequently, the probability that an oxygen molecule in an air bubble will "find" and come into contact with a sodium sulfide molecule within the black liquor is greatly increased.

In addition to creating a much larger surface area of air contacting the black liquor, the rapid rotary motion of the rotor within the housing in conjunction with the turbulent cavitation activity in the cavitation zones causes the very small air bubbles that are created to be distributed through the black liquor in an extremely uniform manner. This further increases the probability that oxygen molecules within the air bubbles will come into contact with sodium sulfide molecules within the black liquor. Such uniform distribution of air bubbles throughout the liquid has been confirmed by observing the electric current drawn by an electric motor driving the rotor. When air bubbles are introduced into the flow of black liquor, this current quickly reaches an equilibrium point, where it remains extremely constant for a given flow of air into the black liquor. If the air bubbles were not uniformly distributed, the current drawn by the motor would fluctuate as the motor encountered pockets of more and less uniformly distributed bubbles within the black liquor. Thus, uniform current draw confirms the uniform distribution of bubbles throughout the black liquor.

Finally, the violent cavitation activity within the cavitation zone imparts sufficient energy to the mixture to break or loosen the molecular van der Waals attractions between the molecules within the black liquor. This results in a breaking up of molecule clusters within the black liquor thereby freeing sodium sulfide molecules that may be surrounded and shielded by other molecules within the liquor. These freed molecules are then available to come into contact with an oxygen molecule within an air bubble and thereby be oxidized. Therefore, the methodology of this invention also affects the black liquor on a molecular level to enhance and increase the percentage of sodium sulfide molecules within the black liquor that are oxidized.

After the black liquor has been treated and its environmentally hazardous compounds oxidized as described, it flows out of the chamber 15 along with residual air bubbles through the outlet port 35. At this point, the excess air within the black liquor quickly begins to agglomerate into larger and larger air bubbles, which naturally separate from the black liquor due to their buoyancy. The oxidized black liquor, which contains substantially smaller quantities of sodium sulfide than with prior art mixing processes, may be used as a fuel in the usual way without producing unacceptable levels of environmental toxins as described above.

FIG. 2 illustrates in a simplified schematic form the application of the present invention for oxidizing black liquor in an industrial setting within the pulp and paper industry. A hydrosonic mixer 11 according to the present invention is powered by a conventional electric motor 46, which spins or rotates the rotor within the mixer at a rapid rate. A holding tank 41 contains a volume of untreated black liquor 42. The untreated black liquor 42 is pumped from the

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holding tank 41 by a pump 43 to the inlet ports 47 of the hydrosonic mixer 11. Air is supplied to and entrained in the form of bubbles within the flow of black liquor through the gas supply conduit 28. The volume of air supplied to the flow of black liquor is controllable with a valve 44. As the black liquor/air bubble mixture passes through the hydrosonic mixer 11, environmentally harmful compounds within the black liquor are oxidized and rendered inert as described above. The treated black liquor and excess air 34 is then delivered to a reservoir tank 48, where excess air 51 within the black liquor 49 naturally separates by floating to the surface of the black liquor. The excess air can be vented through a vent 52. The remaining black liquor, with its environmentally harmful compounds oxidized, can then be drawn from the reservoir tank 48 through a conduit 53 and valve 54 for subsequent environmentally clean combustion to provide heat where required in the paper making process.

As mentioned above, the methodology of the present invention has been described within the context of oxidizing environmentally harmful compounds within black liquor in the pulp and paper industry. However, the method is equally applicable to a wide variety of industrial processes, some of which are discussed briefly as follows.

1. Oxidation: The term "oxidation" is used throughout this specification to refer to an interaction between two compounds wherein a chemical reaction occurs between the compounds. Oxidation may, as in the case of the oxidation of black liquor discussed above, involve a chemical reaction with oxygen; however, the term "oxidation" as used herein is not intended to be limited to such reactions but rather to include chemical reactions between any elements and/or compounds. Such reactions may include classic oxidation/reduction reactions as well as other types of chemical reactions. The oxidation of environmentally harmful compounds within black liquor in the pulp and paper industry as described above is one example of the application of the present invention for oxidation. Other examples include, but are not limited to, satisfying biological oxygen demand (BOD) and chemical oxygen demand (COD) requirements in the wastewater treatment industry prior to discharging treated wastewater into rivers. In addition to the oxidation of black liquor in the pulp and paper industry, white liquor also may be oxidized for use in bleaching operations. Broadly, the methodology of the present invention is applicable to any situation where one substance is to be mixed with another in such a way that a chemical reaction occurs between the substances or a compound that is an integral part of one or more of the substances.

2. Oxygenation: The mixing of oxygen (or another gas) with a liquid in such a way that the oxygen molecules are trapped and held by van der Waals attractions between the liquid molecules is known as oxygenation. Oxygenation differs from oxidation in that a chemical reaction between the gas and liquid molecules does not occur but, instead, the gas molecules are dissolved in the liquid and loosely held between the liquid molecules. Oxygenation of liquids has applications, for example, in the oxygenation of fuels and fuel oils to enhance burning efficiency in boilers and in the oxygenation of water in aquariums and other aquatic tanks to provide essential oxygen to aquatic animals. Oxygenated drinking water also is becoming popular among many people.

Efficient and effective oxygenation of liquids may be obtained with the method of the present invention in much the same way as oxidation. Air in the form of bubbles is introduced into the liquid to be oxygenated and the mixture is introduced into the hydrosonic mixer, where the air

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bubbles are transformed into millions of microscopic bubbles by cavitation effects. The microscopic air bubbles are then distributed uniformly through the liquid and, at the same time, the van der Waals attractions between the liquid molecules are weakened or broken by the cavitation effects. Subsequently, when the molecules again begin to cluster as a result of van der Waals attractions, air or oxygen molecules become trapped and held within clusters of liquid molecules and the liquid is oxygenated. Because of the exceedingly small air bubbles, uniform distribution, and van der Waals effects, the methodology of the present invention results in substantially enhanced oxygenation efficiency over prior art techniques.

3. Heat Extraction From Stack Gases: In some applications, it is desirable to extract the heat from stack gases emanating from furnaces and boilers and to use the extracted heat in a constructive way such as to heat a liquid. Mixing of the hot stack gases with a liquid is one method of transferring the heat of the stack gases into the liquid. Other methods such as passing the gases through a heat exchanger immersed in the liquid also have been used to accomplish this goal. The methodology of the present invention is particularly efficient at transferring heat from hot stack gases to a liquid. In such an application, the hot stack gases are introduced into a stream of liquid and the mixture is directed into the hydrosonic mixer. Here, as in oxidation and oxygenation, the stack gases are distributed uniformly throughout the liquid. Because of the large composite area of contact between the hot stack gas bubbles and the liquid, the heat of the gases is transferred efficiently and effectively to the liquid. Thus, heat energy otherwise lost to the atmosphere or transferred inefficiently with prior art techniques is transferred to and heats the liquid. The stack gases (now cooled) and the liquid (now heated) can then be separated and the stack gases released to the atmosphere. Accordingly, the method of the invention may be applied in a highly efficient and effective process for recapturing and using the heat of stack gases.

4. Dissolved Air Flotation: Dissolved air flotation is a process whereby air (or another gas) is introduced into and mixed in a liquid having suspended particles that need to be separated from the liquid. Generally, the air molecules and small bubbles of air, once mixed in the liquid, attach themselves to the suspended particles within the liquid causing them to float to the surface of the liquid where they can be removed. Dissolved air flotation may be used, for example, in the pulp and paper industry to remove suspended fibers from a liquid. The method of the present invention is well suited for dissolved air flotation. In practicing the method for this application, air is introduced into the liquid and the mixture is directed to the hydrosonic mixer. There, the air bubbles are reduced to microscopic size and distributed uniformly through the liquid, resulting in a higher probability that the air bubbles will attach themselves to the suspended particles to be separated from the liquid. Upon leaving the hydrosonic mixture, the liquid may be directed to a separation tank, where the particles float to the surface under the buoyant influence of the attached air bubbles and can be skimmed or otherwise removed.

5. Mixing Of Dissimilar Fluids: In many industrial applications, it is desirable to mix two dissimilar fluids such as, for example, water and oil, to form an emulsion. The making of caulking compounds and the formation of oil and water emulsions are examples of such applications. The method of the present invention may be applied efficiently to form emulsions from dissimilar fluids. More specifically, one of the fluids, water for example, is introduced into a

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stream of the other fluid, oil for example, and the mixture is fed to the hydrosonic mixer. In the mixer, the cavitation bubbles and shock waves created mechanically by the rotating rotor impact the liquids breaking them down into extremely small units or droplets. The droplets are then distributed uniformly together by the violent agitation within the mixer. This, in conjunction with the weakening or breaking of the van der Waals attractions between fluid molecules, results in the small droplets of the fluids being attracted to and "holding onto" one another to form a tight and uniform emulsion.

6. Separation Of Emulsified Liquids: In some cases, it is desirable to separate a tight emulsion into its component liquids. For example, in the oil industry, oil from a well may take the form of an oil/water emulsion and it is necessary to separate the water from the oil prior to further processing. One way to accomplish this is to mix a gas, such as methane, with the oil/water emulsion to interfere with the van der Waals attraction between the oil and water molecules. The method of the present invention has been found to accomplish this task in a very reliable and efficient way. More specifically, the gas, methane for example, is introduced into a stream of the emulsion and the mixture is directed to the hydrosonic mixer according to the invention. In the mixer, the van der Waals attractions between the molecules of the two fluids in the emulsion are broken down and the methane gas is reduced to microscopic bubbles and distributed uniformly throughout the emulsion. Under these conditions, the methane gas molecules may attach themselves to one of the liquids preventing it from recombining with the other liquid and thereby separating the emulsion into its constituent components. The separated liquids may then be drawn off separately by conventional methods.

7. Atomization of Fuels to Enhance Burning

Atomizing a fuel before it is burned enhances the combustion efficiency of the fuel by exposing a significantly larger surface area of the fuel to heat and oxidizer. The mixing methodology of the present invention is well suited to the atomization of fuels. In an atomization application, a gas, which may be an oxygen containing gas to enhance burning further or may be an inert gas, is mixed with the fuel in the hydrosonic mixer according to the invention. The gas is broken down in to microscopic bubbles and dispersed uniformly throughout the fluid. This mixture may then be delivered to a boiler or other device in which the fuel is to be burned, where it is ejected as an atomized spray comprising very tiny droplets. The burning efficiency of the fuel is therefore enhanced.

The invention has been described and disclosed herein in terms of preferred specific examples, embodiments, and methodologies that represent the best mode known to the inventors of carrying out the invention. However, the invention is not limited to these illustrative embodiments and many other applications and variations may be made within the scope of the invention. Further, devices with configurations other than that illustrated herein for mechanically inducing cavitation effects used in the method of the invention are possible, the illustrated devices being only exemplary illustrations. In general, any mechanical apparatus capable of producing the cavitation effects described herein for purposes of mixing two dissimilar fluids is considered to be within the scope of the invention. These and many other additions, deletions, and modifications to the disclosed embodiments and methodologies may well be made by those of skill in the art without departing from the spirit and scope of the invention as set forth in the claims.

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What is claimed is:

1. A method of oxidizing a molecular compound in black liquor that is to be burned as a fuel, the molecular compound, when not oxidized, generating environmental toxins upon burning of the black liquor, said method comprising the steps of:

- (a) introducing an oxidizer into the black liquor to create a mixture;
- (b) introducing the mixture into a chamber having a rotor formed with a plurality of irregularities, the irregularities on the rotor inducing cavitation within the mixture to reduce the oxidizer to a large number of relatively small units; and
- (c) distributing the relatively small units of oxidizer uniformly throughout the mixture.

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2. A method of oxidizing a molecular compound in black liquor as claimed in claim 1 and wherein the molecular compound to be oxidized is sodium sulfide.

3. A method of oxidizing a molecular compound in black liquor as claimed in claim 2 and wherein the oxidizer is oxygen.

4. A method of oxidizing a molecular compound in black liquor as claimed in claim 3 and wherein step (a) comprises introducing air into the black liquor, the air containing oxygen.

5. A method of oxidizing a molecular compound in black liquor as claimed in claim 1 and where in step (c) the relatively small units of oxidizer are distributed throughout the mixture by the action of the rotor within the chamber.

* * * * *

EXHIBIT “F”



US010220365B2

(12) **United States Patent**
Mancosky

(10) **Patent No.:** **US 10,220,365 B2**
(45) **Date of Patent:** **Mar. 5, 2019**

(54) **METHOD AND APPARATUS FOR HYDROGENATING SUBSTANCES USING CONTROLLED MECHANICALLY INDUCED CAVITATION**

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(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 54 days.

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B01F 7/00 (2006.01)

(52) **U.S. Cl.**
CPC **B01J 19/008** (2013.01); **B01F 7/00816** (2013.01); **B01J 19/006** (2013.01); **B01J 19/18** (2013.01); **B01J 19/1806** (2013.01); **B01J 2219/00049** (2013.01); **B01J 2219/00768** (2013.01); **B01J 2219/00777** (2013.01)

(58) **Field of Classification Search**
CPC B01J 19/006; B01J 19/008; B01J 19/18; B01J 19/1806; B01J 2219/00049; B01J 2219/00768; B01J 2219/00777; C07C 5/00; C07C 5/02; C07C 29/04; C07C 29/106; C07C 209/36; C07C 45/34; C07C 67/08; C07C 45/39; C07C 51/265; C07C 211/46; C07C 211/50; C07C 31/202; C07C 31/205; C07C 31/207; C07C 31/22; C07C 31/225; C07C 35/14; C07C 47/06; C07C 49/04; C07C 49/08; C07C 63/06; C07C 69/003

See application file for complete search history.

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(57) **ABSTRACT**

A method and apparatus of inducing chemical reactions such as hydrogenation of a fluidized substance includes mixing hydrogen with the substance, passing the mixture through a cavitation zone, and inducing intense shockwaves in the cavitation zone by continuously generating high energy cavitation events within the mixture. In one embodiment, the treatment zone is the interior of a controlled cavitation reactor. Interior surfaces of the reactor may be coated with a catalyst required for the desired chemical reaction so that the catalyst need not be added directly to the mixture and need not be recovered after the reaction is complete.

5 Claims, 1 Drawing Sheet

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FIG. 1

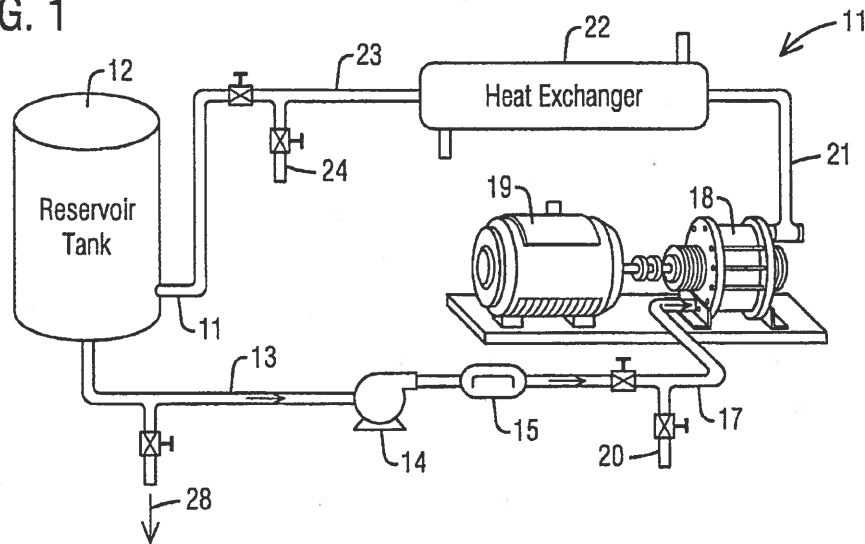
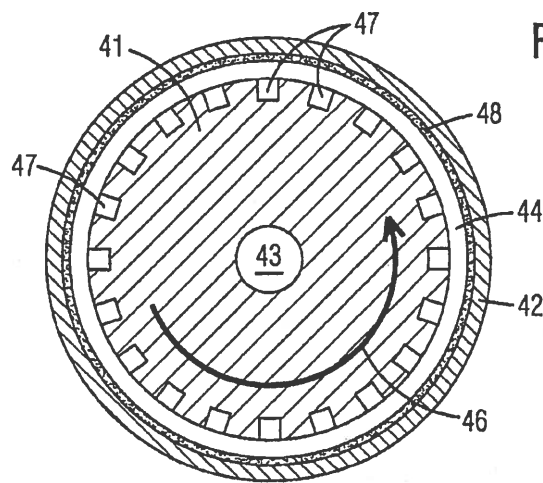


FIG. 2

Equipment	*(k _L a), 1/s
Mechanically Agitated Tank	0.02 - 0.2
Controller Cavitation Reactor	0.90 - 5.2

*(k_La), 1/s; Mass Transfer Coefficient

FIG. 3



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METHOD AND APPARATUS FOR HYDROGENATING SUBSTANCES USING CONTROLLED MECHANICALLY INDUCED CAVITATION

REFERENCE TO RELATED APPLICATION

Priority is hereby claimed to the filing date of U.S. provisional patent application 62/140,968 filed on Mar. 31, 2015, the contents of which are hereby incorporated by reference.

TECHNICAL FIELD

This invention relates generally to reacting hazardous and/or highly reactive gases in a small volume utilizing a high mass transfer controlled cavitation reactor to enhance reaction efficiency and safety. In one embodiment, the invention relates to hydrogenation of substances within the reactor with or without a catalyst.

BACKGROUND

Industrial chemical reactions are carried out in a wide array of manufacturing operations. Such reactions often involve or liberate highly reactive material or hazardous material that can present a significant safety risk. Examples of industrial chemical reactions that fall into this category include the production of ethylene oxide, fluorination, and hydrogenation to name a few. Hydrogenation is a chemical reaction between molecular hydrogen (H_2) and another compound or element, usually in the presence of a catalyst such as nickel, palladium or platinum.

The largest scale application of hydrogenation is for the processing of vegetable oils in the food industry. Typical vegetable oils are derived from polyunsaturated fatty acids (containing more than one carbon-carbon double bond). Their partial hydrogenation reduces most but not all, of these carbon-carbon double bonds. The degree of hydrogenation is controlled by restricting the amount of hydrogen, reaction temperature and time, and the catalyst. In the case of hydrogenation, hydrogen is a highly reactive molecule and can be dangerous and hazardous if not contained and controlled very carefully.

Industrial chemical reactions such as hydrogenation commonly are carried out in large reaction vessels and may involve mixing H_2 with a substance to be hydrogenated in the presence of a catalyst and stirring the mixture. This process can be hazardous at least in part because of the large volume of material being reacted at one time and the highly reactive nature of the H_2 . Such reactions also often require expensive catalysts that are not always completely recoverable or are expensive to recover after the reaction is complete.

A need exists for a method and apparatus for carrying out chemical reactions such as hydrogenation involving highly reactive or hazardous material that is safe, fast, easily scalable to handle commercial volumes reactants and products, highly effective and efficient, and that does not require that a catalyst or catalysts be recovered after the reaction is complete. It is to the provision of such a method and apparatus that the present invention is primarily directed.

SUMMARY

Briefly described, a method is disclosed for conducting a chemical reaction involving dangerous or highly volatile or

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reactive chemicals or gasses in a manner that is far safer and far more efficient than traditional methods. For example, hydrogen can be a highly reactive, volatile, and dangerous gas when used in industrial chemical reactions such as hydrogenation. This invention includes conducting such reactions in a modified small volume, high mass transfer, controlled cavitation reactor such as the Shockwave Power Reactor available from Hydro Dynamics LLC of Rome, Ga., USA. Versions of such a reactor are disclosed in U.S. Pat. Nos. 8,465,642; 8,430,968; 7,507,014; 7,360,755; and 6,627,784, all owned by the assignee of the present application and all incorporated herein by reference. Such reactors are characterized by a cavitation zone within which reactions occur that is extremely small in volume in comparison to the volume of a standard batch reaction tank. The chemical reaction takes place in this cavitation zone, aided by intense shock waves produced by high energy cavitation events.

By conducting dangerous and/or hazardous chemical reactions such as hydrogenation within such a small volume rather than in large reaction tanks as in the past, the risk is reduced by a significant amount. Due to the mass transfer efficiency of the controlled cavitation reactor, reaction efficiency also can be improved from efficiencies of batch reaction techniques. If a catalyst or catalysts are needed to catalyze the desired chemical reactions, the catalyst can be coated on the internal surfaces of the reactor, thereby catalyzing desired reactions without the need to recover the catalysts after the reaction is complete. The relatively small controlled cavitation reactor can be attached as a side stream of an already existing batch process or incorporated as part of a continuous process. The invention will be better appreciated upon review of the detailed description set forth below when taken in conjunction with the accompanying drawing figures, which are briefly described as follows.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 illustrates an embodiment of a controlled cavitation reactor for carrying out the method of the invention.

FIG. 2 is a chart that illustrates the high mass transfer ability of the controlled cavitation reactor compared to a conventional batch reaction system.

FIG. 3 is a simplified cross sectional view illustrating interior components of a controlled cavitation reactor with a catalyst coated internal surface.

DETAILED DESCRIPTION

Many chemical reactions and gas-liquid mixing process involve conditions that can be dangerous, hazardous to health, and volatile. Further, many such chemical reactions and gas-liquid mixing involve components or end products of high value so that it is critical that the process be carried out with confidence without jeopardizing the end product. Reactions and mixing under one or more of the following conditions can qualify as hazardous.

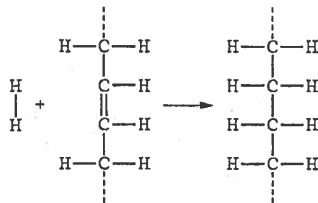
1. Reacting or Mixing at High Temperatures
2. Reacting or Mixing at High Pressures
3. Reactions that Require Expensive Gasses or a High Cost Catalyst
4. Highly Exothermic Reactions
5. Reactions or Mixing Resulting in High Value End Product or a Product of extreme importance

Hydrogenation is one example of high value reaction and is one of the most widely used industrial processes, particularly in the food industry. Hydrogenation involves the mix-

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ing of hydrogen gas (H_2) into substances (usually oils) at elevated temperatures and pressures. Catalysts often are added to catalyze the desired chemical reaction wherein most of the carbon-carbon double bonds of the oil molecules are reduced according to the below reaction.



Hydrogenation is a strongly exothermic reaction. In the hydrogenation of vegetable oils and fatty acids, for example, the heat released is about 25 kcal per mole (105 kJ/mol), sufficient to raise the temperature of the oil by 1.6-1.7° C. per iodine number drop.

Hydrogenation using traditional batch reaction processes usually is hindered by poor mixing. Hydrogenation is carried out on a micro liter scale to thousands of gallons of substance per minute, depending upon the industry and application. Due at least in part to the poor mixing achieved in traditional processes, hydrogenation is normally carried out with excess hydrogen and excess catalyst, either in a packed bed or entrained as a powder, to make up for the poor mixture. The catalyst is often a high value metal such as platinum or palladium and therefore can be very expensive. It is therefore important to capture and recover as much catalyst as possible without damage it in order to maximize economics. This can be an inefficient and expensive process.

The present invention comprises a method of mixing hazardous gases like hydrogen with liquids and/or solids to achieve desired chemical reactions with safety and high efficiency. The method is carried out in a continuous process wherein a mixture of gas and liquid and/or solid is passed through the cavitation zone of a controlled cavitation reactor. Within the cavitation zone, highly energetic shock waves generated by cavitation events permeate the mixture. These shock waves, in turn, break up gas bubbles to form bubbles of extremely small size and distribute these uniformly through the mixture. This results in much more complete mixing of the gas with the liquid and greatly increased surface area contact between the molecules of gas and liquid. The ultimate result is a chemical reaction such as hydrogenation that is substantially more efficient and complete than is possible with traditional batch reaction processes.

With more complete mixing using mechanically induced cavitation in the reactor, less hydrogen can be used at lower temperatures and pressures resulting in a significant cost savings. Final product quality can also be higher due to more complete reactions and higher yields. The method of this invention also can have a safety benefit should ignition occur. This is due at least in part to an inherent much smaller mixing volume compared to traditional large stirred reaction vessels. The invention will be described within the context of hydrogenation applications wherein hydrogen gas is mixed with a substance to be hydrogenated. It will be understood, however, that the invention is not limited to this exemplary embodiment but may be used to improve the efficiency of other reactions involving hazardous or expensive materials.

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FIG. 1 illustrates in simplified schematic form a controlled cavitation reactor system usable to carry out the methodology of the present invention. Such a reactor is described in detail in the incorporated references, and so will be describe only generally here. The reactor 11 comprises a reservoir tank 12 for holding a liquid or other substance to be hydrogenated. The liquid is drawn from the reservoir tank 12 through conduit 13 by a pump 14 and delivered through a flow meter 16 and conduit 17 to a controlled cavitation reactor 18. A gas inlet 20 communicates with the conduit 17 for introducing hydrogen to the liquid. The controlled cavitation reactor 18 generally comprises a cylindrical housing having an internally mounted cylindrical rotor. A space is defined between the outer peripheral surface of the rotor and the inner peripheral surface of the housing and this space is referred to as the cavitation zone. Cavitation-inducing structures such as radial bores are formed in or on the outer peripheral surface of the rotor. The rotor is rotated within the housing by an electric motor 19.

The liquid is pumped through conduit 17 toward the cavitation reactor, and hydrogen (or another appropriate gas or gas mixture) is introduced into the liquid flow through gas inlet 20. The mixture of liquid and hydrogen then flows through the cavitation zone of the controlled cavitation reactor. As the rotor is rotated at a high rate, continuous cavitation events are induced in the liquid within the radial bores of the rotor. This, in turn, produces highly energetic shock waves caused by the continuously collapsing cavitation bubbles in the bores and the shock waves propagate through the liquid-gas mixture in the cavitation zone.

The energetic shock waves induce a number of effects in the gas-liquid mixture. More specifically, the hydrogen bubbles within the mixture are broken up by the shock waves into millions of tiny bubbles of hydrogen. This, in turn, greatly increases the total surface area of hydrogen molecules contacting molecules of the liquid. Further, the tiny bubbles of hydrogen are distributed uniformly throughout the mixture as the mixture flows through the cavitation zone. The uniform distribution of tiny hydrogen bubbles ensures that concentrations of bubbles and voids without bubbles in the mixture are eliminated, thereby maximizing surface area contact between the liquid and hydrogen molecules.

After treatment in the reactor 18, the liquid-gas mixture flows through conduit 21 and may flow through a heat exchanger 22 to remove unwanted heat induced by the energy of cavitation. The cooled liquid then flows through conduit 23 and inlet 26 back to the reservoir tank 12, where any remaining hydrogen gas and other gasses migrate out of the liquid. The liquid may be circulated through the cavitation reactor as many times as desired to obtain a desired level of hydrogenation of the liquid. Then, it can be extracted as a hydrogenated end product, as indicated by arrow 28. Alternately, the system of FIG. 1 may be installed upstream of or downstream of a traditional hydrogenation in a hybrid system for hydrogenating a substance.

In a preferred embodiment, a liquid and hydrogen gas are passed through and mixed within the controlled cavitation reactor to induce hydrogenation reactions. These reactions usually require a catalyst, which as mentioned above can be an expensive material that must be recovered after the reactions are complete. One aspect of the present invention is that internal components of the cavitation reactor such as surfaces of the rotor and/or surfaces of the housing or stator are coated with the needed catalyst. Another benefit of the reactor is that a liquid or mixture within its cavitation zone is in constant motion, resulting in a very high refresh rate at

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internal surfaces of the reactor. In fact, it has been found that at any point on the inner housing surface, the refresh rate or the number of times a given small volume of reactants encounter a catalyst coated surface may be as high as several thousand times per second.

A desired chemical reaction, which inherently occurs at a catalyst coated surface, benefits from this very high surface refresh rate. More of the tiny and uniformly distributed gas bubbles and liquid contact the catalyst coated surface, which improves exposure to the catalyst, improves selectivity, and improves the rate of chemical hydrogenation reactions. As a result, surface area requirements are decreased as are operational costs, capital costs, and footprint requirements.

With the great surface refreshing induced in the cavitation zone, it becomes possible to coat the internal surfaces of the controlled cavitation reactor with amenable catalysts. This eliminates the need to add catalysts directly to the liquid, and thus eliminates post processing wherein the catalyst must be removed from the hydrogenated product. Cost savings with less catalyst used and/or the elimination of process steps can be substantial. Perhaps just as salient, continuous hydrogenation at commercial volumes is easily achieved with greatly reduced risks of exposure to hazardous materials and ignition with the method and apparatus of the present invention.

Through process intensification it is also possible to treat a substance continuously or have only a small volume of a batch loop where all three components (gas, liquid, and catalyst) meet and have the potential for maximum exothermic and runaway reactions. This serves to allow similar total production, but minimize the amount of fluid being treated at any one time, minimizing many of the inherent risks. A cooling jacket can be disposed around the reactor to remove excess heat generated in the hydrogenation reaction processes, thereby further enhancing safety.

If a powdered catalyst is used there is still benefit since the intense pressure fluctuations induced by cavitation can drive the fluid and gas deep inside the catalyst particles allowing for maximum catalyst surface area contact, minimum catalyst usage and enhanced reactions.

FIG. 2 is a table comparing the mass transfer efficiency of hydrogenation with a traditional mechanically agitated tank to the mass transfer efficiency of hydrogenation using the method and apparatus of the present invention. As can be seen, the mass transfer efficiency with the present invention is orders of magnitude greater than the results using traditional agitated reaction tank techniques.

FIG. 3 shows the interior components of a controlled cavitation reactor for carrying out the method of this invention. The reactor comprises a cylindrical internal rotor 41 rotatably mounted within a cylindrical housing 42. A shaft 43 is rotated by an electric motor to spin the rotor 41 at a high rate of rotation within the housing 42, as indicated by arrow 46. The peripheral surface of the rotor 41 is spaced from the interior peripheral surface of the housing 42 to define a cavitation zone 44 between the two. Cavitation inducing structures such as radial bores 47 are formed in the peripheral surface of the rotor 41. As the rotor 41 spins in the housing 42 with a fluidized substance in the cavitation zone, cavitation events are continuously created within the radial bores 47. These events induce highly energetic shock waves that propagate through the fluid within the cavitation zone 44, as described above.

For purposes of hydrogenation according to the present invention, a catalyst material 48 is coated on one or more interior surfaces of the controlled cavitation reactor. In this example, the catalyst is coated on the inner peripheral

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surface of the cylindrical housing. However other or additional surfaces also can be coated with the catalyst if desired. The methodology of the invention also can be carried out with powdered catalyst entrained in the fluid, but this is not the most efficient technique since the catalyst will have to be recovered from the hydrogenated substance following hydrogenation.

As discussed above, a mixture of a substance to be hydrogenated and hydrogen gas is pumped through the cavitation zone of the controlled cavitation reactor. The highly energetic shock waves induced in the mixture creates millions of tiny hydrogen bubbles and distributes them evenly throughout the mixture. The shock waves also create an extremely high refresh rate of the mixture at the surface of the catalyst coating 48. The combination of these factors in addition to high mass transfer induces hydrogenation of the substance with very high effectiveness and efficiency. Since the catalyst is coated on an interior surface of the controlled cavitation reactor, there is no need to recover catalyst from the hydrogenated substance after hydrogenation.

The invention has been described herein in terms of preferred embodiments and methodologies considered by the inventor to represent the best mode of carrying out the invention. More specifically, use of the invention for inducing hydrogenation reactions has been used as a primary example. However, the invention is not limited to hydrogenation, but is applicable to inducing any suitable chemical reaction. It will be understood by the skilled artisan that a wide gamut of additions, deletion, and modifications of the exemplary embodiments, both subtle and gross, may well be made without departing from the spirit and scope of the invention, which is delineated only by the claims.

What is claimed is:

1. A method of hydrogenating a fluidized substance comprising the steps of:

- (a) obtaining a controlled cavitation reactor having a cylindrical rotor rotatably mounted within a cylindrical housing with a cavitation zone defined between the peripheral surface of the rotor and the internal peripheral surface of the housing, the cylindrical rotor having bores formed through its peripheral surface, the controlled cavitation reactor having no structures that protrude into the cavitation zone;
- (b) generating a flow of the fluidized substance;
- (c) introducing hydrogen gas into the flow of the fluidized substance to form a mixture;
- (d) passing the mixture through the cavitation zone of the controlled cavitation reactor and rotating the rotor to induce cavitation events in the bores of the rotor thereby resulting in shock waves that propagate through the mixture within the cavitation zone, the shock waves breaking up the hydrogen gas into small bubbles and distributing the bubbles evenly throughout the mixture;
- (e) allowing the mixture to remain in the cavitation zone for a time sufficient to induce the chemical reaction between the hydrogen gas and the fluidized substance with a mass transfer coefficient between about 0.9 and about 5.2 $k_L a$, 1/s; and
- (f) recovering the substance resulting from the chemical reaction from the controlled cavitation reactor.

2. The method of claim 1 further comprising the step of exposing the mixture to a catalyst within the cavitation zone of the controlled cavitation reactor.

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3. The method of claim 2 wherein the step of exposing the mixture to a catalyst comprises introducing catalyst into the flow of the mixture.

4. The method of claim 2 wherein the step of exposing the mixture to a catalyst comprises coating at least one interior surface of the controlled cavitation reactor with the catalyst. 5

5. The method of claim 4 wherein the step of coating at least one interior surface of the controlled cavitation reactor comprises coating the inner peripheral surface of the housing with the catalyst. 10

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(12) **United States Patent**
Mancosky

(10) **Patent No.:** **US 10,220,365 B2**

(45) **Date of Patent:** **Mar. 5, 2019**

(54) **METHOD AND APPARATUS FOR
HYDROGENATING SUBSTANCES USING
CONTROLLED MECHANICALLY INDUCED
CAVITATION**

(71) Applicant: **Hydro Dynamics, Inc., Rome, GA
(US)**

(72) Inventor: **Douglas G. Mancosky, White, GA
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B01J 2219/00049 (2013.01); **B01J 2219/00768**
(2013.01); **B01J 2219/00777** (2013.01)

(58) **Field of Classification Search**
CPC **B01J 19/006**; **B01J 19/008**; **B01J 19/18**;
B01J 19/1806; **B01J 2219/00049**; **B01J**
2219/00768; **B01J 2219/00777**; **C07C**
5/00; **C07C 5/02**; **C07C 29/04**; **C07C**
29/106; **C07C 209/36**; **C07C 45/34**; **C07C**
67/08; **C07C 45/39**; **C07C 51/265**; **C07C**
211/46; **C07C 211/50**; **C07C 31/202**;
C07C 31/205; **C07C 31/207**; **C07C 31/22**;
C07C 31/225; **C07C 35/14**; **C07C 47/06**;
C07C 49/04; **C07C 49/08**; **C07C 63/06**;
C07C 69/003

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Primary Examiner — Sharon Pregler

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(57) **ABSTRACT**

A method and apparatus of inducing chemical reactions such as hydrogenation of a fluidized substance includes mixing hydrogen with the substance, passing the mixture through a cavitation zone, and inducing intense shockwaves in the cavitation zone by continuously generating high energy cavitation events within the mixture. In one embodiment, the treatment zone is the interior of a controlled cavitation reactor. Interior surfaces of the reactor may be coated with a catalyst required for the desired chemical reaction so that the catalyst need not be added directly to the mixture and need not be recovered after the reaction is complete.

5 Claims, 1 Drawing Sheet

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FIG. 1

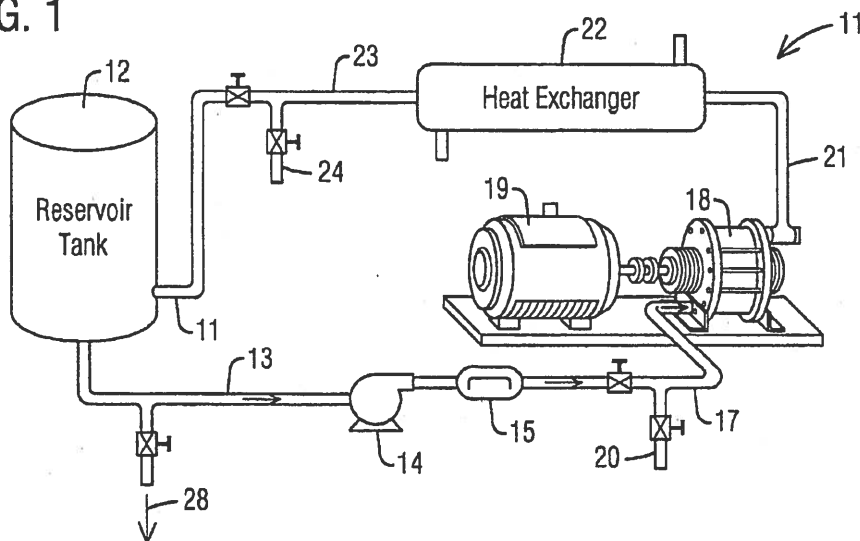
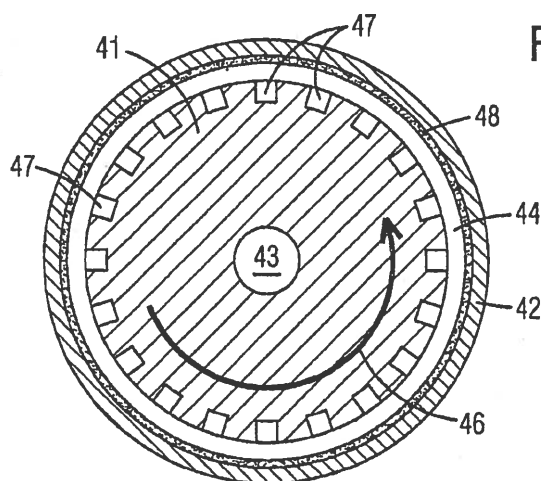


FIG. 2

Equipment	* $(k_L a)$, 1/s
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* $(k_L a)$, 1/s; Mass Transfer Coefficient

FIG. 3



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Industrial chemical reactions such as hydrogenation commonly are carried out in large reaction vessels and may involve mixing H_2 with a substance to be hydrogenated in the presence of a catalyst and stirring the mixture. This process can be hazardous at least in part because of the large volume of material being reacted at one time and the highly reactive nature of the H_2 . Such reactions also often require expensive catalysts that are not always completely recoverable or are expensive to recover after the reaction is complete.

A need exists for a method and apparatus for carrying out chemical reactions such as hydrogenation involving highly reactive or hazardous material that is safe, fast, easily scalable to handle commercial volumes reactants and products, highly effective and efficient, and that does not require that a catalyst or catalysts be recovered after the reaction is complete. It is to the provision of such a method and apparatus that the present invention is primarily directed.

SUMMARY

Briefly described, a method is disclosed for conducting a chemical reaction involving dangerous or highly volatile or

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reactive chemicals or gasses in a manner that is far safer and far more efficient than traditional methods. For example, hydrogen can be a highly reactive, volatile, and dangerous gas when used in industrial chemical reactions such as hydrogenation. This invention includes conducting such reactions in a modified small volume, high mass transfer, controlled cavitation reactor such as the Shockwave Power Reactor available from Hydro Dynamics LLC of Rome, Ga., USA. Versions of such a reactor are disclosed in U.S. Pat. Nos. 8,465,642; 8,430,968; 7,507,014; 7,360,755; and 6,627,784, all owned by the assignee of the present application and all incorporated herein by reference. Such reactors are characterized by a cavitation zone within which reactions occur that is extremely small in volume in comparison to the volume of a standard batch reaction tank. The chemical reaction takes place in this cavitation zone, aided by intense shock waves produced by high energy cavitation events.

By conducting dangerous and/or hazardous chemical reactions such as hydrogenation within such a small volume rather than in large reaction tanks as in the past, the risk is reduced by a significant amount. Due to the mass transfer efficiency of the controlled cavitation reactor, reaction efficiency also can be improved from efficiencies of batch reaction techniques. If a catalyst or catalysts are needed to catalyze the desired chemical reactions, the catalyst can be coated on the internal surfaces of the reactor, thereby catalyzing desired reactions without the need to recover the catalysts after the reaction is complete. The relatively small controlled cavitation reactor can be attached as a side stream of an already existing batch process or incorporated as part of a continuous process. The invention will be better appreciated upon review of the detailed description set forth below when taken in conjunction with the accompanying drawing figures, which are briefly described as follows.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 illustrates an embodiment of a controlled cavitation reactor for carrying out the method of the invention.

FIG. 2 is a chart that illustrates the high mass transfer ability of the controlled cavitation reactor compared to a conventional batch reaction system.

FIG. 3 is a simplified cross sectional view illustrating interior components of a controlled cavitation reactor with a catalyst coated internal surface.

DETAILED DESCRIPTION

Many chemical reactions and gas-liquid mixing process involve conditions that can be dangerous, hazardous to health, and volatile. Further, many such chemical reactions and gas-liquid mixing involve components or end products of high value so that it is critical that the process be carried out with confidence without jeopardizing the end product. Reactions and mixing under one or more of the following conditions can qualify as hazardous.

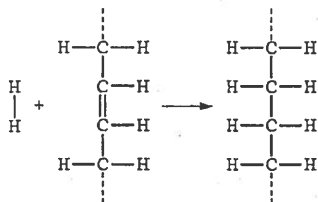
1. Reacting or Mixing at High Temperatures
2. Reacting or Mixing at High Pressures
3. Reactions that Require Expensive Gasses or a High Cost Catalyst
4. Highly Exothermic Reactions
5. Reactions or Mixing Resulting in High Value End Product or a Product of extreme importance

Hydrogenation is one example of high value reaction and is one of the most widely used industrial processes, particularly in the food industry. Hydrogenation involves the mix-

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ing of hydrogen gas (H_2) into substances (usually oils) at elevated temperatures and pressures. Catalysts often are added to catalyze the desired chemical reaction wherein most of the carbon-carbon double bonds of the oil molecules are reduced according to the below reaction.



Hydrogenation is a strongly exothermic reaction. In the hydrogenation of vegetable oils and fatty acids, for example, the heat released is about 25 kcal per mole (105 kJ/mol), sufficient to raise the temperature of the oil by 1.6-1.7° C. per iodine number drop.

Hydrogenation using traditional batch reaction processes usually is hindered by poor mixing. Hydrogenation is carried out on a micro liter scale to thousands of gallons of substance per minute, depending upon the industry and application. Due at least in part to the poor mixing achieved in traditional processes, hydrogenation is normally carried out with excess hydrogen and excess catalyst, either in a packed bed or entrained as a powder, to make up for the poor mixture. The catalyst is often a high value metal such as platinum or palladium and therefore can be very expensive. It is therefore important to capture and recover as much catalyst as possible without damage it in order to maximize economics. This can be an inefficient and expensive process.

The present invention comprises a method of mixing hazardous gases like hydrogen with liquids and/or solids to achieve desired chemical reactions with safety and high efficiency. The method is carried out in a continuous process wherein a mixture of gas and liquid and/or solid is passed through the cavitation zone of a controlled cavitation reactor. Within the cavitation zone, highly energetic shock waves generated by cavitation events permeate the mixture. These shock waves, in turn, break up gas bubbles to form bubbles of extremely small size and distribute these uniformly through the mixture. This results in much more complete mixing of the gas with the liquid and greatly increased surface area contact between the molecules of gas and liquid. The ultimate result is a chemical reaction such as hydrogenation that is substantially more efficient and complete than is possible with traditional batch reaction processes.

With more complete mixing using mechanically induced cavitation in the reactor, less hydrogen can be used at lower temperatures and pressures resulting in a significant cost savings. Final product quality can also be higher due to more complete reactions and higher yields. The method of this invention also can have a safety benefit should ignition occur. This is due at least in part to an inherent much smaller mixing volume compared to traditional large stirred reaction vessels. The invention will be described within the context of hydrogenation applications wherein hydrogen gas is mixed with a substance to be hydrogenated. It will be understood, however, that the invention is not limited to this exemplary embodiment but may be used to improve the efficiency of other reactions involving hazardous or expensive materials.

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FIG. 1 illustrates in simplified schematic form a controlled cavitation reactor system usable to carry out the methodology of the present invention. Such a reactor is described in detail in the incorporated references, and so will be describe only generally here. The reactor 11 comprises a reservoir tank 12 for holding a liquid or other substance to be hydrogenated. The liquid is drawn from the reservoir tank 12 through conduit 13 by a pump 14 and delivered through a flow meter 16 and conduit 17 to a controlled cavitation reactor 18. A gas inlet 20 communicates with the conduit 17 for introducing hydrogen to the liquid. The controlled cavitation reactor 18 generally comprises a cylindrical housing having an internally mounted cylindrical rotor. A space is defined between the outer peripheral surface of the rotor and the inner peripheral surface of the housing and this space is referred to as the cavitation zone. Cavitation-inducing structures such as radial bores are formed in or one the outer peripheral surface of the rotor. The rotor is rotated within the housing by an electric motor 19.

The liquid is pumped through conduit 17 toward the cavitation reactor, and hydrogen (or another appropriate gas or gas mixture) is introduced into the liquid flow through gas inlet 20. The mixture of liquid and hydrogen then flows through the cavitation zone of the controlled cavitation reactor. As the rotor is rotated at a high rate, continuous cavitation events are induced in the liquid within the radial bores of the rotor. This, in turn, produces highly energetic shock waves caused by the continuously collapsing cavitation bubbles in the bores and the shock waves propagate through the liquid-gas mixture in the cavitation zone.

The energetic shock waves induce a number of effects in the gas-liquid mixture. More specifically, the hydrogen bubbles within the mixture are broken up by the shock waves into millions of tiny bubbles of hydrogen. This, in turn, greatly increases the total surface area of hydrogen molecules contacting molecules of the liquid. Further, the tiny bubbles of hydrogen are distributed uniformly throughout the mixture as the mixture flows through the cavitation zone. The uniform distribution of tiny hydrogen bubbles ensures that concentrations of bubbles and voids without bubbles in the mixture are eliminated, thereby maximizing surface area contact between the liquid and hydrogen molecules.

After treatment in the reactor 18, the liquid-gas mixture flows through conduit 21 and may flow through a heat exchanger 22 to remove unwanted heat induced by the energy of cavitation. The cooled liquid then flows through conduit 23 and inlet 26 back to the reservoir tank 12, where any remaining hydrogen gas and other gasses migrate out of the liquid. The liquid may be circulated through the cavitation reactor as many times as desired to obtain a desired level of hydrogenation of the liquid. Then, it can be extracted as a hydrogenated end product, as indicated by arrow 28. Alternately, the system of FIG. 1 may be installed upstream of or downstream of a traditional hydrogenation in a hybrid system for hydrogenating a substance.

In a preferred embodiment, a liquid and hydrogen gas are passed through and mixed within the controlled cavitation reactor to induce hydrogenation reactions. These reactions usually require a catalyst, which as mentioned above can be an expensive material that must be recovered after the reactions are complete. One aspect of the present invention is that internal components of the cavitation reactor such as surfaces of the rotor and/or surfaces of the housing or stator are coated with the needed catalyst. Another benefit of the reactor is that a liquid or mixture within its cavitation zone is in constant motion, resulting in a very high refresh rate at

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internal surfaces of the reactor. In fact, it has been found that at any point on the inner housing surface, the refresh rate or the number of times a given small volume of reactants encounter a catalyst coated surface may be as high as several thousand times per second.

A desired chemical reaction, which inherently occurs at a catalyst coated surface, benefits from this very high surface refresh rate. More of the tiny and uniformly distributed gas bubbles and liquid contact the catalyst coated surface, which improves exposure to the catalyst, improves selectivity, and improves the rate of chemical hydrogenation reactions. As a result, surface area requirements are decreased as are operational costs, capital costs, and footprint requirements.

With the great surface refreshing induced in the cavitation zone, it becomes possible to coat the internal surfaces of the controlled cavitation reactor with amenable catalysts. This eliminates the need to add catalysts directly to the liquid, and thus eliminates post processing wherein the catalyst must be removed from the hydrogenated product. Cost savings with less catalyst used and/or the elimination of process steps can be substantial. Perhaps just as salient, continuous hydrogenation at commercial volumes is easily achieved with greatly reduced risks of exposure to hazardous materials and ignition with the method and apparatus of the present invention.

Through process intensification it is also possible to treat a substance continuously or have only a small volume of a batch loop where all three components (gas, liquid, and catalyst) meet and have the potential for maximum exothermic and runaway reactions. This serves to allow similar total production, but minimize the amount of fluid being treated at any one time, minimizing many of the inherent risks. A cooling jacket can be disposed around the reactor to remove excess heat generated in the hydrogenation reaction processes, thereby further enhancing safety.

If a powdered catalyst is used there is still benefit since the intense pressure fluctuations induced by cavitation can drive the fluid and gas deep inside the catalyst particles allowing for maximum catalyst surface area contact, minimum catalyst usage and enhanced reactions.

FIG. 2 is a table comparing the mass transfer efficiency of hydrogenation with a traditional mechanically agitated tank to the mass transfer efficiency of hydrogenation using the method and apparatus of the present invention. As can be seen, the mass transfer efficiency with the present invention is orders of magnitude greater than the results using traditional agitated reaction tank techniques.

FIG. 3 shows the interior components of a controlled cavitation reactor for carrying out the method of this invention. The reactor comprises a cylindrical internal rotor 41 rotatably mounted within a cylindrical housing 42. A shaft 43 is rotated by an electric motor to spin the rotor 41 at a high rate of rotation within the housing 42, as indicated by arrow 46. The peripheral surface of the rotor 41 is spaced from the interior peripheral surface of the housing 42 to define a cavitation zone 44 between the two. Cavitation inducing structures such as radial bores 47 are formed in the peripheral surface of the rotor 41. As the rotor 41 spins in the housing 42 with a fluidized substance in the cavitation zone, cavitation events are continuously created within the radial bores 47. These events induce highly energetic shock waves that propagate through the fluid within the cavitation zone 44, as described above.

For purposes of hydrogenation according to the present invention, a catalyst material 48 is coated on one or more interior surfaces of the controlled cavitation reactor. In this example, the catalyst is coated on the inner peripheral

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surface of the cylindrical housing. However other or additional surfaces also can be coated with the catalyst if desired. The methodology of the invention also can be carried out with powdered catalyst entrained in the fluid, but this is not the most efficient technique since the catalyst will have to be recovered from the hydrogenated substance following hydrogenation.

As discussed above, a mixture of a substance to be hydrogenated and hydrogen gas is pumped through the cavitation zone of the controlled cavitation reactor. The highly energetic shock waves induced in the mixture creates millions of tiny hydrogen bubbles and distributes them evenly throughout the mixture. The shock waves also create an extremely high refresh rate of the mixture at the surface of the catalyst coating 48. The combination of these factors in addition to high mass transfer induces hydrogenation of the substance with very high effectiveness and efficiency. Since the catalyst is coated on an interior surface of the controlled cavitation reactor, there is no need to recover catalyst from the hydrogenated substance after hydrogenation.

The invention has been described herein in terms of preferred embodiments and methodologies considered by the inventor to represent the best mode of carrying out the invention. More specifically, use of the invention for inducing hydrogenation reactions has been used as a primary example. However, the invention is not limited to hydrogenation, but is applicable to inducing any suitable chemical reaction. It will be understood by the skilled artisan that a wide gamut of additions, deletion, and modifications of the exemplary embodiments, both subtle and gross, may well be made without departing from the spirit and scope of the invention, which is delineated only by the claims.

What is claimed is:

1. A method of hydrogenating a fluidized substance comprising the steps of:

- (a) obtaining a controlled cavitation reactor having a cylindrical rotor rotatably mounted within a cylindrical housing with a cavitation zone defined between the peripheral surface of the rotor and the internal peripheral surface of the housing, the cylindrical rotor having bores formed through its peripheral surface, the controlled cavitation reactor having no structures that protrude into the cavitation zone;
- (b) generating a flow of the fluidized substance;
- (c) introducing hydrogen gas into the flow of the fluidized substance to form a mixture;
- (d) passing the mixture through the cavitation zone of the controlled cavitation reactor and rotating the rotor to induce cavitation events in the bores of the rotor thereby resulting in shock waves that propagate through the mixture within the cavitation zone, the shock waves breaking up the hydrogen gas into small bubbles and distributing the bubbles evenly throughout the mixture;
- (e) allowing the mixture to remain in the cavitation zone for a time sufficient to induce the chemical reaction between the hydrogen gas and the fluidized substance with a mass transfer coefficient between about 0.9 and about 5.2 $k_L a$, 1/s; and
- (f) recovering the substance resulting from the chemical reaction from the controlled cavitation reactor.

2. The method of claim 1 further comprising the step of exposing the mixture to a catalyst within the cavitation zone of the controlled cavitation reactor.

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3. The method of claim 2 wherein the step of exposing the mixture to a catalyst comprises introducing catalyst into the flow of the mixture.

4. The method of claim 2 wherein the step of exposing the mixture to a catalyst comprises coating at least one interior surface of the controlled cavitation reactor with the catalyst. 5

5. The method of claim 4 wherein the step of coating at least one interior surface of the controlled cavitation reactor comprises coating the inner peripheral surface of the housing with the catalyst. 10

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EXHIBIT “G”



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5 YEARS IN THE MAKING

We are the creators of the Noah strain, developed with the intention of cultivating a portfolio of unique micro-cannabinoids and terpenes

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WHO IS APOTHIO?

Apothio is a team of world class
doctors, researchers, chemists,
engineers, and agronomists on a

mission to pioneer “Food as Medicine”.

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We have proudly and naturally developed our own Hemp genetics in the Mojave Desert of California.

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Acres Planted

Average Plant Size (ft)

Million Grams of CBD

Years in the making

WHAT WE DO

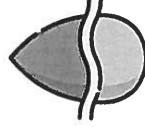
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Apothio is dedicated to pioneering technology that redefines standards for safety and toxicology, objectively demonstrating the efficacy of our hemp-derived cannabinoid formulations, and collaborating with patients across the country to develop the most unique and customized plant based formulations on the market.



Unique Hemp Genetics

Apothio is a research pioneer in the natural development of Hemp genetics with unique cannabinoid and terpene profiles designed for superior outcomes.



Proprietary Extraction

Apothio is developing cutting edge technology that replaces toxic and volatile solvents from the extraction process, and replaces it with water.



Data-Driven Formulations

Apothio utilizes proprietary whole plant extraction technology that uses water as the solvent, which produces a safe and compliant water soluble extract.



Research Institute

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Apothio is partnered with institutions of higher education to conduct workforce development, educational programs, and research outcomes with our patients.

OUR METHODOLOGY

Our whole plant oil CBD oil is much more than just CBD.

The cannabis plant contains thousands of synergistic compounds, all of which all work together in what we refer to metaphorically as a biochemical symphony.

Extraction methodologies today filter out these synergistic molecules to achieve the “purest” form of CBD, leaving most consumers with a full price ticket to a symphony with only one instrum

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Our hemp-derived, whole plant extraction methodology embraces each and every biochemical component from both male and female hemp plants, ultimately giving you the effect that nature intended.

Snapshot of the biochemical composition

CBD	CBDa	CBDv	CBG	THCva
			CBC	THCv

- An array of beneficial cannabinoids such as CBG, THCv, THCva, CBD, CBDa, CBDv, CBDva, CBC, and less than 0.3% THC
- Beneficial terpenes such as Humulene, Caryophyllene, Myrcene, Pinene, Ocimene, Terpinene, and Limonene

- Flavonoids such as Silymarin, Orientin, Quercetin, Kaempferol, and Cannafavin

- Chlorophyll and phytol

- Fats and waxes

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As Nature Intended

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Dr. Trent Jones, Apothio CEO

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